QUARTERLY PROGRESS REPORT NUMBER ONE

INVESTIGATION OF STRUCTURAL PROPERTIES OF FIBER GLASS FILAMENT-WOUND PRESSURE VESSELS AT CRYOGENIC TEMPERATURES

OCTOBER 1963
DOUGLAS REPORT SM-45762

MISSILE & SPACE SYSTEMS DIVISION
DOUGLAS AIRCRAFT COMPANY, INC.
SANTA MONICA CALIFORNIA

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PREPARED AND SUBMITTED BY: J.M. TOTH, JR. Oct,
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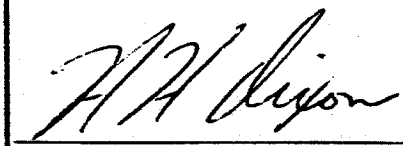

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DOUGLAS MISSILE & SPACE SYSTEMS DIVISION

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FOREWORD

This report was prepared by the Douglas Aircraft Company, Inc., Missiles and Space Systems Division under NASA Contract NAS 3-2562. This investigation was initiated by Lewis Research Center of NASA to determine the structural properties of fiber glass filament-wound pressure vessels at cryogenic temperatures. The work is being administered by Mr. James R. Barber, Chemical Rocket Systems Division, NASA/Lewis Research Center, 21000 Brookpark Road, Cleveland, 35, Ohio.

This is the first quarterly progress report and covers work done between 1 July 1963 and 1 October 1963.

Included among those who cooperated in the research and the preparation of the report were: J. M. Toth, Jr., Investigation Director, Advance Space Technology: R. Harvey, W. H. Kimberly, R. B. Lantz, B. E. Newnam, A. C. Rawuka, L. M. Roseland, T. T. Sakurai, Materials Research and Production Methods; E. R. Charhut, Structural Test; Aerospace Systems Engineering: R. W. Bowman, Engineering Research and Development Laboratories: and D. W. Yockey, Reliability.

ABSTRACT

This is an interim report on a development program for the investigation of structural properties of fiber glass filament-wound pressure vessels at cryogenic temperatures. The general approach and preliminary results are described.

The following tasks were completed during the period of this report:

1. Uniaxial mechanical properties of the organic and metallic candidate liner materials.

2. Thermal contraction properties of the candidate liners and the three epoxy resin/fiber glass composites.

3. Unstressed room temperature permeability tests of the candidate liners.

4. Uniaxial cyclic tests of resin/fiber glass composites at -320° F and -423° F; selection of a preliminary best resin system.

5. Fabrication of room temperature biaxial test specimens.

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SECTION I

INTRODUCTION

This document is the first quarterly progress report for Contract No. NAS 3-2562 sponsored by NASA/Lewis Research Center, Cleveland, Ohio. The project is a 16-month development program divided into four phases. The scheduling of the four phases of the program has been arranged to permit the required "closed loop" between design, test, and fabrication.

The Phase I Effort includes the orderly investigation and recommendation of liner materials and resin/fiber glass composites suitable for use with either liquid nitrogen or liquid hydrogen. A previous Douglas Independent Research and Development program had demonstrated the feasibility of nickel and Mylar liners with their corresponding advantages and disadvantages. These included considerations of permeability, compatibility with the laminate under fatigue and ultimate loads, and weight. It is not obvious, however, that nickel and Mylar are the best materials in their representative families. It is therefore necessary to determine the properties of other liner materials that may have a great potential. These liner materials include, in addition to Mylar and nickel, Tedlar, H-film, polyurethane film, glass flakes, copper, and silver.

The basic structural properties of fiber glass vessel designs must also be investigated. Test data show cracking of the resins at low values of strain in a cryogenic environment. Therefore, three different resin/fiber glass composites are being evaluated in conjunction with S-994 glass filaments. The investigation will determine liner compatibility and material properties by means of coupons and small cylinders. The properties include tensile yield and ultimate strength, modulus of elasticity, ultimate elongation, coefficient of thermal contraction, density, and liner permeability to gaseous and liquid nitrogen and hydrogen. Review of the data will provide information for the selection of a liner and a resin/fiber glass system.

Two 18-inch by 24-inch filament-wound pressure vessels will be fabricated for testing with liquid hydrogen.

Twenty 18-inch diameter by 24-inch long pressure vessels will be fabricated in Phase II for testing with either liquid nitrogen or liquid hydrogen. The fabrication schedule will be paced by the testing schedule since there is a possibility of revising the vessel design, as the test program progresses, in order to provide an acceptable failure mode.

The Phase III effort consists of burst and cycling tests of small-scale fiberglass filament-wound pressure vessels at liquid nitrogen temperature. And, the Phase IV effort consists of burst and cycling tests of small-scale fiber glass filament-wound pressure vessels at liquid hydrogen temperature.

The objectives of Phases III and IV are:

1. Determination of burst strengths at -320° F and -423°F.
2. Determination of number of cycles required to fail the filament-wound pressure vessels at 60%, 70%, 80%, and 90% of ultimate pressure determined in (1) above.
3. Determination of the tank circumferential and longitudinal strains as a function of vessel pressure for each cycle.
4. Establishment of the efficiency parameter PV/W using burst pressure for P and total vessel weight for W.

SECTION 2

PHASE I - LINER AND RESIN SYSTEM EVALUATION

In Phase I, the objective is to design and develop small-scale fiber glass filament-wound pressure vessels to contain cryogenic fluids. To reach this goal, Douglas is conducting an orderly investigation of liner materials and fiber glass resin composites for cryogenic applications. The materials and processes being used in the small-scale fiber glass tanks are also suitable for use in full-scale tanks.

A previous Douglas program had demonstrated the feasibility of certain types of integral metal and plastic liners. It is believed that the integral liners have a greater potential than bladders for propellant tanks. Integral liners reduce assembly problems and permit more reliable installation of internal tank fittings and attachments. The main problem is the strain compatibility with the fiber glass tank walls.

The selected liner must possess the following characteristics:

1. Impermeable to cryogenic fluids
2. Almost impermeable to gases from cryogenic fluids
3. Chemically inert with either fluid or gas.

A determination of the properties and accompanying processes of certain potentially good liner materials is being made; these include metallic and plastic films. The metallic films include electrochemically deposited nickel, copper, and silver, the plastic films include Mylar, Tedlar, H-film, polyurethane film, and glass flakes. Three resin/fiber glass composites are being evaluated in conjunction with S-994 glass filaments.

During this phase, liner compatibility and material properties have been determined by means of test coupons and small cylinders. The properties of the liner and fiber glass include tensile yield and ultimate strength, modulus of elasticity, ultimate elongation, cyclic resistance, coefficient

of thermal contraction and density. The permeability of the liner to gaseous and liquid nitrogen and hydrogen is also being determined. These data will be the basis for recommending a liner and fiber glass/resin system. Two 18-inch by 24-inch filament-wound pressure vessels will be designed and fabricated for testing with liquid hydrogen.

The approach to Phase I is based on the following considerations and is being carried out as described below and illustrated in Figure 2-1.

CONSIDERATIONS

1. Liner Selection - The choice of the liner primarily depends on the compatibility of the liner-fiber glass composite under ultimate and fatigue stresses in a cryogenic environment. The permeability, weight, and manufacturing requirements of the liner have a major influence. The type of resin system is anticipated to have a secondary effect.

2. Resin System Selection - The resin system will be primarily selected on the basis of its response to fatigue stresses in a cryogenic environment. Variations in ultimate strength of the composite are probably not more than 10% for suitable resin systems. The ultimate strength properties of the liner-resin/fiber glass composite are relatively independent of the liner.

METHOD OF INVESTIGATION

1. Screening of Liners - To provide design information, uniaxial tests have been conducted on all candidate liners to determine (1) mechanical properties* at room temperature, -320°F, and -423°F, (2) unstressed sample permeability of nitrogen and hydrogen gas at room temperature, and (3) coefficient of thermal contraction and density.

2. Selection of Initial Resin System - Three resins have been evaluated on the basis of desired cyclic characteristics. (The field of selection has been narrowed significantly based on the considerations given in Section 2.2). Uniaxial tensile coupon tests at -423°F indicated the selection of a suitable initial resin system for investigating the liners. The other two resins are being set aside for further evaluation until later in the program. Coefficient of thermal contraction tests have been conducted on the three resin systems.

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* Mechanical properties include yield and ultimate tensile strength, modulus of elasticity, and ultimate elongation.

PHASE I – LINER AND RESIN SYSTEM EVALUATION

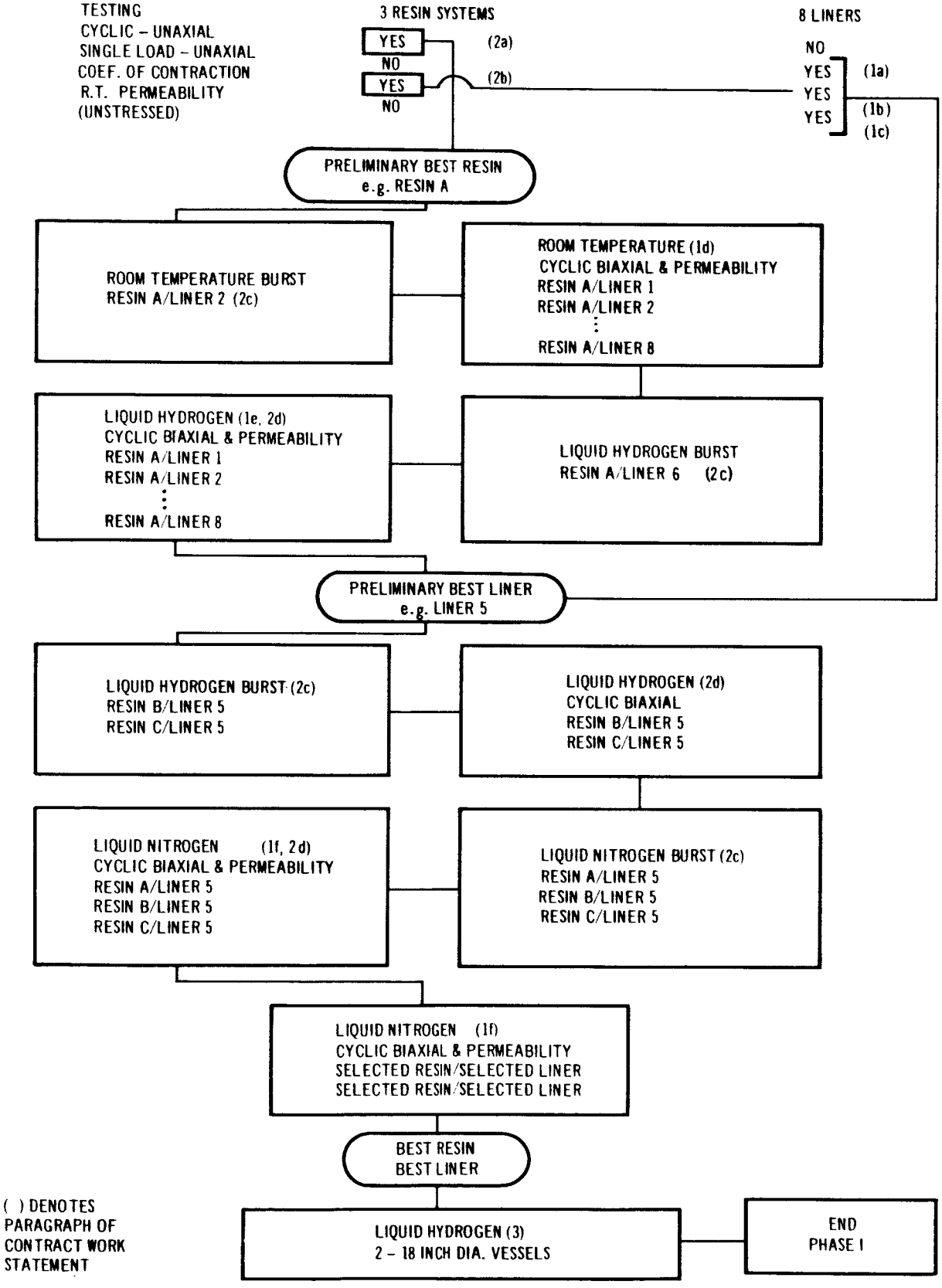


FIGURE 2-1

3. Determination of Initial Resin/Fiber Glass System Mechanical Properties - The mechanical properties at room temperature and -423°F of the chosen resin-fiber glass composite will be determined by a biaxial specimen. (A polyurethane liner was used for the room temperature test and a "battleship" electrodeposited nickel liner will probably be used for the -423°F test.)

4. Determination of Liner Room - Temperature Permeability Rate - Room-temperature hydrogen gas permeability rates of the candidate liners will be measured in a 60% of ultimate biaxial stress field. Upon complete acquisition of permeability data, the specimens will be cycled at 60% of ultimate to failure.

5. Determination of Liner LH_2 Permeability Rates - Liquid hydrogen permeability rates of the candidate liners will be measured in a 60% of ultimate biaxial stress field. Upon complete acquisition of permeability data, the specimens will be cycled at 60% of ultimate to failure .

6. Selection of Preliminary Liner - From the information obtained in Steps 1 to 5, the best preliminary liner will be selected.

7. Determination of Resin/Fiber Glass System Mechanical Properties - Using the best liner, a determination will be made of the mechanical properties at -423°F of the two resin systems set aside in Step 2 and at -320°F of all three resin systems. These tests will be made with biaxial specimens.

8. Determination of Resin/Fiber Glass System Mechanical Properties - Using the best liner, Douglas will determine the biaxial permeability, and cycling characteristics at -423°F of the two systems set aside in Step 2 and at -320°F of all three resin systems at 60% of ultimate.

9. Additional Evaluation - Several additional selected liner-resin combinations will be tested for 60% ultimate permeability rates and cycling resistance at -320°F and 423°F.

10. Selection of Liner-Resin - From Steps 6 to 9, all of the design information will have been obtained, and it will be possible to select the best liner and fiber glass/resin composite for use in the two vessels.

11. Manufacture of Two Vessels - Two 18-inch diameter by 24-inch long vessels will be fabricated.

2.1 LINER MATERIAL INVESTIGATION

Douglas has selected eight candidate materials as potential liners:

1. Mylar
2. Tedlar
3. H-Film
4. Polyurethane Film
5. Glass Flakes
6. Nickel
7. Copper
8. Silver

This section presents the justification for their selection.

These candidate materials were selected on the basis of an evaluation program at Douglas and available information in the literature. Some of the criteria for candidate selection were:

1. The structural fiber glass/composite wall will strain approximately 3 to 6% in liquid hydrogen, therefore, the ultimate strain of the liner should be within this range in order to effectively utilize the composite strength.

2. The coefficient of thermal contraction of the liner must be compatible with the structural composite wall since the structure will be subjected to cyclic thermal loads.

3. The material must be impermeable to the fluid and have a low comparative gas permeability rate.

Materials included in the Douglas preliminary IR&D program and literature search but rejected from further evaluation are listed in Table 2-I with the reasons for rejection.

The use of metallics solves one problem but poses another. There is no doubt that with suitable fabrication techniques, a metallic liner can be made which will be impermeable to liquid and gaseous hydrogen (References 1, 7, 8, and 9). But the strain compatibility with metallics is a serious problem, as demonstrated in the Douglas preliminary program. Metallics such as nickel, copper, beryllium-copper, stainless steel, lead, and aluminum became debonded and elongated. Some, such as aluminum and lead, cracked at points of high strain.

Table 2-1

REJECTED LINER MATERIALS

Material	Reason for Rejection
Organics	
Aluminized Mylar	Aluminum cracking and brittleness when immersed in liquid hydrogen (Reference 1).
Saran	Brittleness and cracking when subjected to cyclic strain in liquid nitrogen (Reference 1).
Kynar	Brittleness and cracking when immersed in liquid hydrogen (Reference 1).
Aclar	Brittleness and cracking when subjected to cyclic strain in liquid hydrogen (Reference 1).
Epoxy Laminate	High ambient-temperature hydrogen gas permeability (Reference 1).
Butyl Rubber	High ambient-temperature hydrogen gas permeability and brittleness at cryogenic temperature (Reference 1).
Dacron-Mylar	Essentially the same as Mylar, with added problems of Dacron. Rejected in favor of straight Mylar. (Reference 1).
Dacron/Aluminum-Mylar/Dacron	High ambient-temperature hydrogen gas permeability (Reference 1).
GRS Hydropol Elastomer	High ambient-temperature hydrogen gas permeability (Reference 1).
DV 1180 Urethane Lacquer	High liquid nitrogen permeability rate and brittleness when stressed in liquid nitrogen (Reference 1).
Teflon (FEP "A" and "C")	High permeability rate and brittleness when subjected to cyclic stress in liquid hydrogen (Reference 1); low elongation in liquid nitrogen (Reference 2); high coefficient of thermal contraction (Reference 3).

Kel-F	Low elongation in liquid nitrogen (Reference 4); high coefficient of thermal contraction Reference 3).
CHR Silicone Rubber	High permeability rate (Reference 1).
Polyethelene	Brittleness and low elongation (Reference 6).
Nylon	Low elongation below -150°F (Reference 5).
Polystyrene	High coefficient of thermal contraction (Reference 3).
Metallics	
Aluminum	Foil cracking when subjected to cyclic strain in liquid hydrogen (specimen bonded to fiber glass composite)(Reference 1).
Aluminum/Mylar/Aluminum	Interlaminar debonding of components and aluminum laminate bonded to fiber glass specimen; cracking when subjected to cyclic strain in liquid hydrogen (Reference 1).
FM 1000/Aluminum/FM 1000	FM 1000 sample had brittleness and cracking during immersion testing in liquid nitrogen (Reference 1).
FEP/Aluminum/FEP	(See note for Teflon under Organics).
Magnesium	Incompatible thermal coefficient of contraction with fiber glass laminate (Reference 3).
Titanium	Incompatible thermal coefficient of contraction with fiber glass laminate (Reference 3).
Zinc	Incompatible thermal coefficient of contraction with fiber glass laminate (Reference 3).
Lead	Foil cracked when subjected to cyclic strain in liquid hydrogen (Reference 1).

Although other materials were suggested at various times (e.g., cork and plastic foams), no literature was available on these materials at cryogenic temperatures and their method of manufacture was subject to numerous variations. It may be possible that a threshold strain could be proposed for use with metallic liners, which would permit straining and subsequent

work hardening above the yield point to be minimized. Cyclic behavior will be demonstrated during the permeability tests.

The reasons for selection of the eight proposed liner materials are given below:

Metals - The electrodeposition method of manufacturing the liner has been successfully accomplished at Douglas. This method provides a jointless liner which can be used for relatively simple shapes such as the biaxial test specimen or complex shapes such as a domed vessel. Its advantages over a welded or jointed liner are obvious. The metal formed by the deposition process is as pore-free as rolled sheet. Plating feasibility tests run on samples of the salt which is used as the winding mandrel for the 18 by 24-inch vessel have shown the plating technique is satisfactory.

The metals selected for use in this investigation are:

Nickel	The material is easily deposited and has the required properties at room temperature. The coefficient of expansion of nickel is very near that of a fiber glass/laminate and the elongation at -423°F is over 20%. Strength is high. (References 3, 10-17).
Copper	This material is also easily deposited and has the required material properties at room temperature. It has lower strength than the nickel but slightly higher elongation. The coefficient of thermal contraction is slightly higher than that of nickel. (References 3, 10, and 12).
Silver	This material is also easily deposited. The room temperature strength (15,000 psi) is rather low compared to the other two selected metals. Elongation at room temperature of 23% and the elongation of 38% in liquid oxygen are its best characteristics. (References 5 and 10.)

Organics - The organic materials are handicapped by permeability to all of the rare gases (References 1, 7, 18, 19, and 20). The probability exists, however, that this permeability could be made sufficiently low so that an organic liner could be utilized. The mechanical property characteristics are more compatible with the fiber glass/composite than the metals.

The organic materials selected for use in this investigation are:

Mylar	This material proved to be the best plastic among those tested in the Douglas preliminary IR&D program. The excellent strength, elongation, and cyclic strain factors evident in preliminary testing were verified by testing results of that program.
Tedlar	This material has been suggested as a candidate with properties similar to Mylar, but having the important added property of being able to be formed. This, of course, would be extremely helpful in contouring the vessel domes. Bonding difficulties are expected to be about the same as those involved with Mylar. Room-temperature hydrogen permeability is reported less than half that of Mylar. (References 21-23).
H-Film	This material is a new DuPont research product with promise of a good potential for cryogenic usage. The strength characteristics are similar to Mylar at room temperature (approximately 20,000 psi). Its most important attribute is the relatively constant elongation over a range from -452°F to room temperature and elevated temperature (50% , 70%, and 90%, respectively). The 50% figure at -452°F is rather high and consequently suspect, but even if the value is only half, 25%, the material possesses unique properties. Due to the newness of this material no bonding techniques at cryogenic temperatures have been reported, but it is expected that the same difficulties are to be expected as with Mylar. (References 22 and 24).

Polyurethane Film	This material is suggested on the basis of recent Douglas test work on cryogenic adhesion. (Reference 25).
Glass Flakes	Flat plate structure attractive as barrier path. This material had initially been scheduled for evaluation as a barrier film for cryogenic foam insulation. The original sample developed cracks due to mishandling. Because another system was developed, the material was abandoned. (References 26 - 28).

Recent work by Mowers (Reference 29), has shown mechanical properties at cryogenic temperatures to be dependent upon crystallinity of the material; more desirable properties being obtained with lower crystallinity. Therefore, the lowest crystallinity materials available in each material family have been secured for test. Crystallinity values of test materials are given in Appendix F.

2.1.1 Mechanical Properties Tests

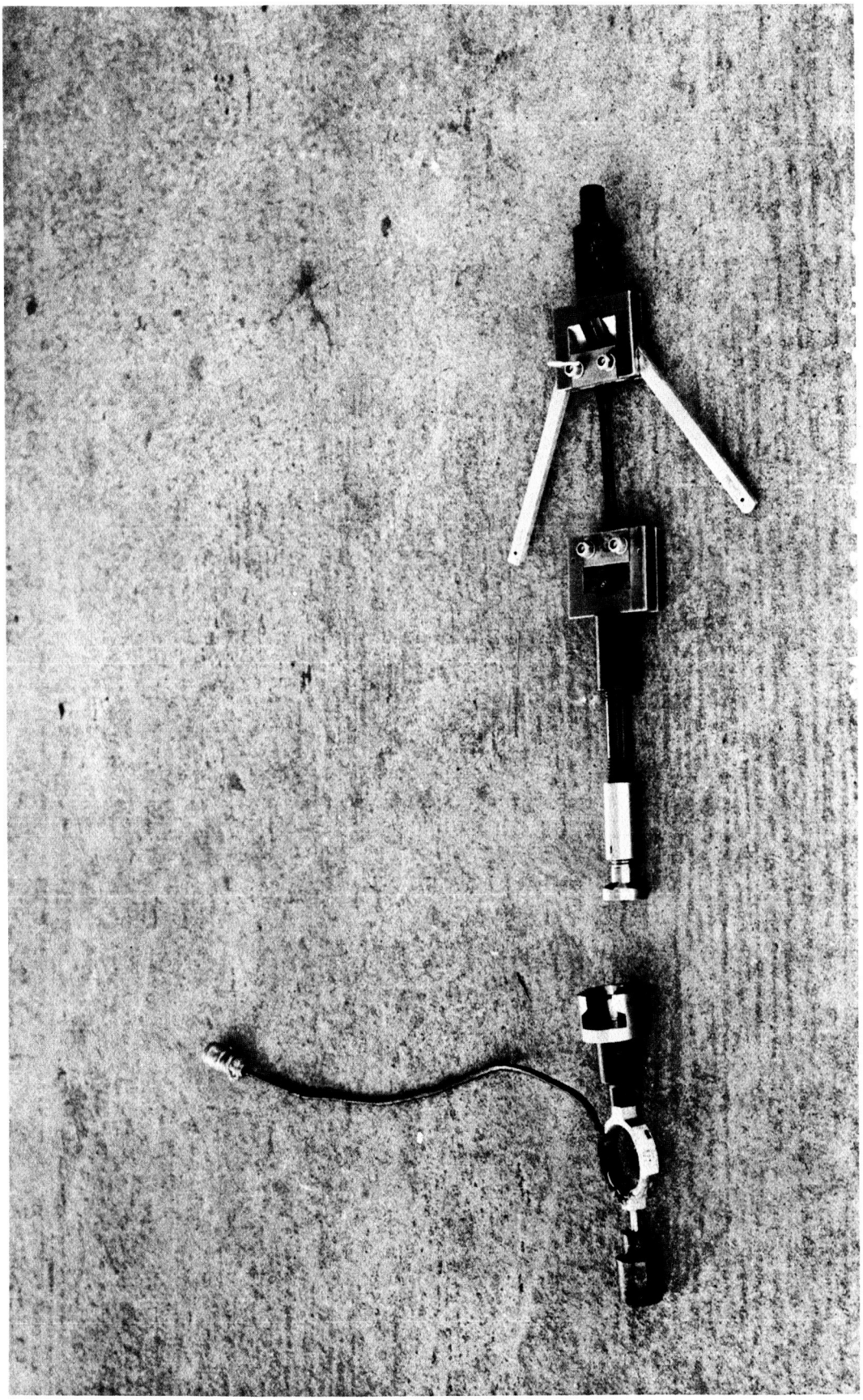
Mechanical properties tests have been made on candidate liners. Uniaxial tensile tests and suitable instrumentation have provided data on the ultimate strength, yield strength, elastic modulus, and ultimate elongation. These tests were run at ambient temperature, -320°F and -423°F.

The thin organic films that were to be evaluated (.001 to .005 inches) excluded any possibility of direct attachment of strain transducers without noticeably affecting the strength of the material and introducing a significant non-axial load factor into the test results. The requirement for evaluation of the materials under study at cryogenic temperatures placed an additional restriction on the test method in that space was limited when testing with the cryogens and there could be no visual observation of the test specimen during the tests.

A special test fixture (Figure 2-2) was fabricated. The same fixture was utilized for all the tests. The grips consisted of two clamp type jaws constructed in a manner such that the line of action of the load train coincided with the axis of the specimen. The jaw faces were lapped together so that the clamping force of the jaws would be evenly distributed across the width of the specimen. The upper and lower jaws were connected by two arms on either side of the specimen and the arms were connected to the jaws by removable pins. The specimens were then mounted in the jaws with the arms connected; the assembly forming one rigid body. In this way no load or twist was placed on the sample. After the two jaws and specimen were installed in the container, the two pins were removed to separate the upper and lower jaws.

Two possible approaches to testing the thin film materials were evident:

1. A straight rectangular specimen would allow direct evaluation of head travel vs. load curves to obtain the desired information.



"JOINED-JAW" TEST GRIPS

FIGURE 2-2

2. A dog-bone shaped specimen would yield fewer failures at the grips and thus produce more reliable data but the analysis of data would be more involved.

The straight rectangular specimens were tested in a preliminary study and were found to have two short comings:

1. A high number of the specimens failed at the grips and made the values for ultimate loads questionable

2. The specimens exhibited large deformations at each jaw. The pull out at the jaws made any head travel measurement conversion into strain unreliable. For these two reasons, the straight rectangular specimen was rejected.

In a preliminary study, modified ASTM standard shaped specimens were tested in the same manner as the straight rectangular specimens and the results indicated that the specimens would break in the reduced section and even though a small amount of local deformation occurred at the jaws, it was decided to utilize this type specimen in order to secure more reliable results. All of the specimens were cut in accordance with modified ASTM Specification D-638-61T. The configuration is shown in Figure 2-3.

The stiffness and strength of the metallic candidate materials permitted the use of mechanical extensometers.

A correction for deformation in the cryostat load train was made and applied to the subsequent crosshead displacements.

It was possible to determine an effective gage length for each dog-bone shaped organic material at room temperature by a time-lapse photographic method described by Smith (Reference 30) and given in the Appendix B. The method permits the conversion of load vs. head travel data into stress strain data. The desired properties could then be read from data plots.

2.1.1.1 Room-Temperature Tests

While the primary interest of this program is in cryogenic temperature,

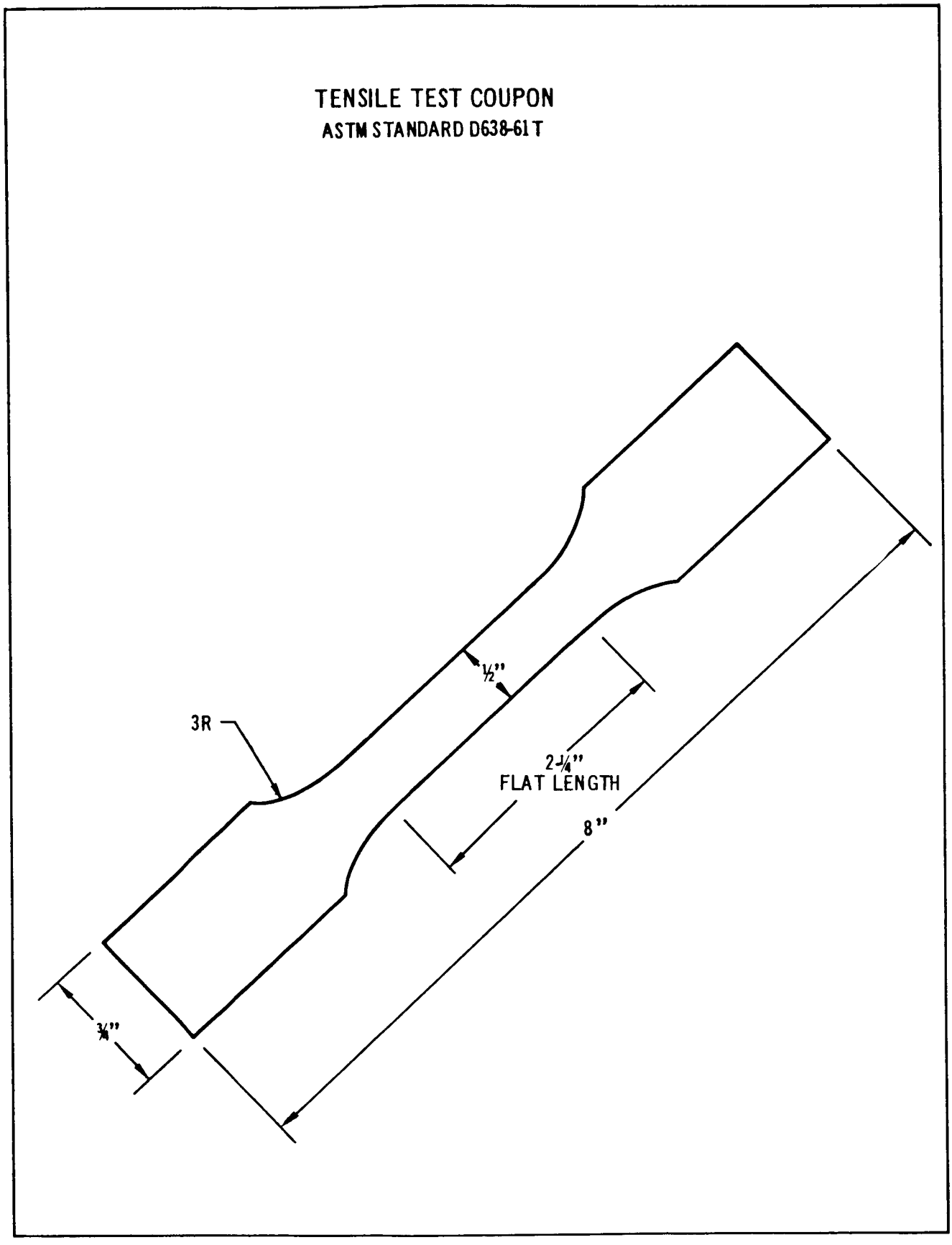

TENSILE TEST COUPON
ASTM STANDARD D638-61T
3R
½"
2¼"
FLAT LENGTH
8"
¾"

FIGURE 2-3

some ambient temperature tests were conducted to provide a basis for comparison. The liner materials involved are all in the thickness range of 0.005 inch. The organic films, in general, are relatively low-strength materials with low moduli and high elongation. These tests were made on an Instron Universal Testing machine, which is ideally suited to low load-high strain testing. Metallic testing was done with extensometers and a 5K Baldwin Universal Testing Machine.

Rates of testing were 0.5 in/minute for all materials, except Tedlar and the polyurethane film: due to the high elongations of these materials, the test rate was 5 in/minute. Load rate for metallics was 1200 psi/min.

2.1.1.2 Tests at -320°F (Liquid Nitrogen)

Organic film tests were made with the Instron machine and a vacuum insulated cryostat, autographic recordings of head travel vs. load were taken. All of the materials were tested at rates of 0.1 in/minute.

The metallic tests were made with the 5K Baldwin machine. Rate of loading was 1200 psi/min.

2.1.1.3 Tests at -423°F (Liquid Hydrogen)

A separate Douglas facility was used for the liquid hydrogen work because of the hazards involved. It was possible to utilize an existing 60K Baldwin Universal Testing Machine in the area, which made it unnecessary to fabricate a special loading frame. A 200-lb capacity load cell was installed in the load train and coupled with an X-Y Moseley Recorder. Two deflectometers were used in the test; one was wired to the X-Y Recorder to give a load vs. head travel curve and the other was used to run the Baldwin Recorder. The strain pacer was used to pace the head rate. The cell and recorder were calibrated directly with class A weights. The deflectometer to the X-Y Moseley Recorder was initially calibrated with a micrometer and checked periodically. The organic materials were tested at 0.1 in/min except for Mylar "A" and the glass flake material, which were tested at 0.5 in/minute.

It had been planned to keep a constant head rate for all temperatures. However, jaw breakage subsequent to the Mylar "A" and glass flake (polyester backing) tests became a factor, so the speed was reduced to 0.10 in/minute.

The metallic tests were made on the 60K Baldwin machine. Rate of loading was **1200 psi/min.**

2.1.1.4 Results

Plots of stress vs. strain and strength vs. temperature are shown in Figures **2-4** through **2-36.** Values for mechanical properties data, e. g., yield point, modulus, etc. have been compiled in tabular form in Appendix B.

For the Organic Films:

All of the materials showed rather high ultimate elongations at room temperature; the polyurethane was highest, 236%, and H-Film was the lowest, 24%. As was expected, elongation became much less at -320°F and -423°F. The highest average elongation at -423°F was that of the glass flake material, 2.71%.

In most cases, strength increased with decrease in temperature. Highest strength in liquid hydrogen was Mylar, 40,000 psi, and lowest was glass flake material, 8,400 psi.

In comparison with published data, the elongation of Mylar at -320°F agrees with that of Miller, (Douglas 5.11% vs. Miller 5.1%) Reference 31 , although ultimate strength does not (Douglas 36,500 psi vs. Miller 44,000 psi). Strength data for Mylar agrees quite well with Mowers, Reference 29 , at all test temperatures, but ultimate elongation is in considerable variance; limited strength data for H-Film agree at the given temperature, (R.T. Douglas 18,000 psi vs. 21,300 psi and -320°F Douglas 24,000 psi vs. 25,000 psi).

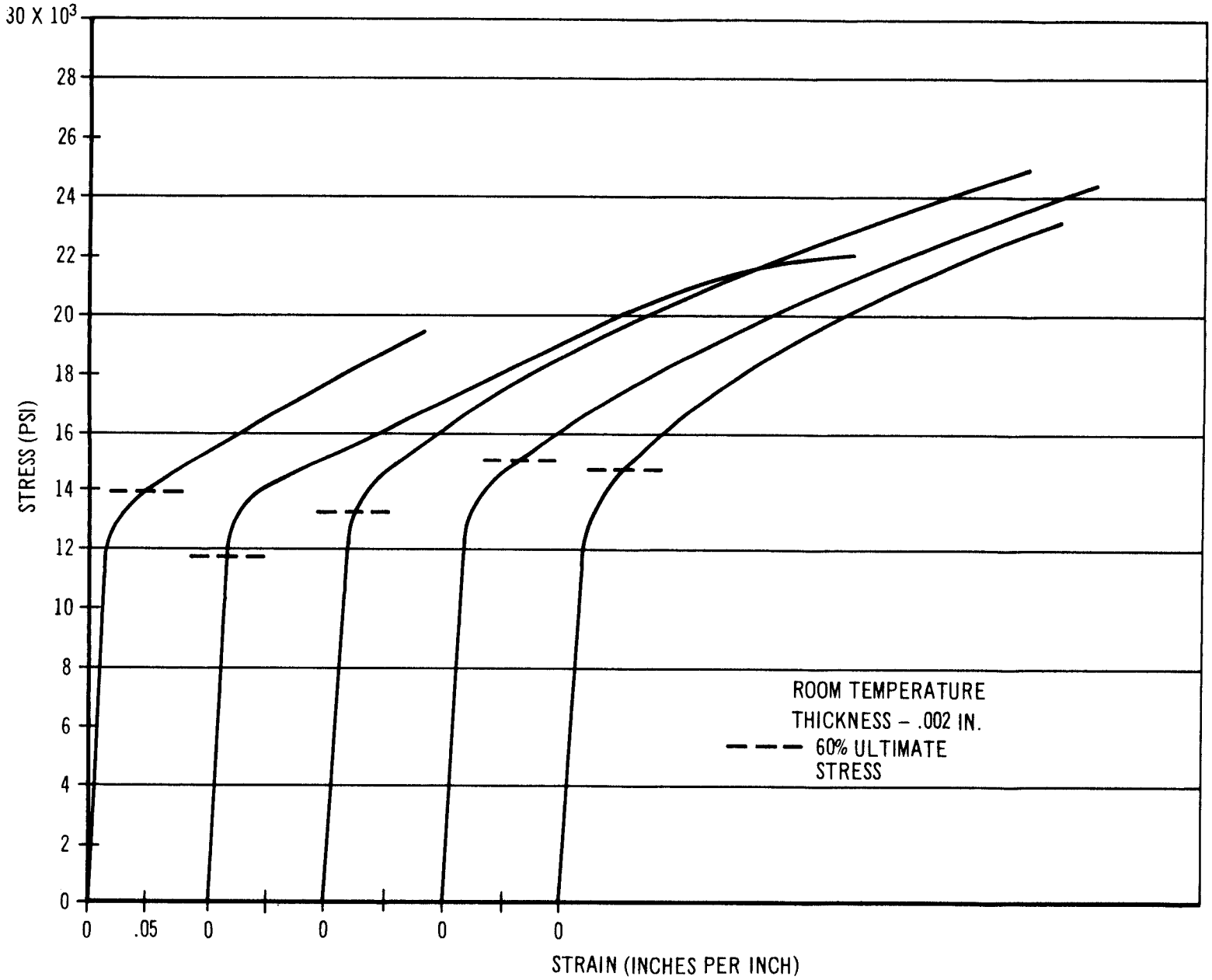

STRESS-STRAIN DIAGRAM FOR MYLAR A FILM
30 X 10³
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
STRESS (PSI)
0
.05
0
0
0
0
STRAIN (INCHES PER INCH)
ROOM TEMPERATURE
THICKNESS – .002 IN.
60% ULTIMATE STRESS

FIGURE 2-4

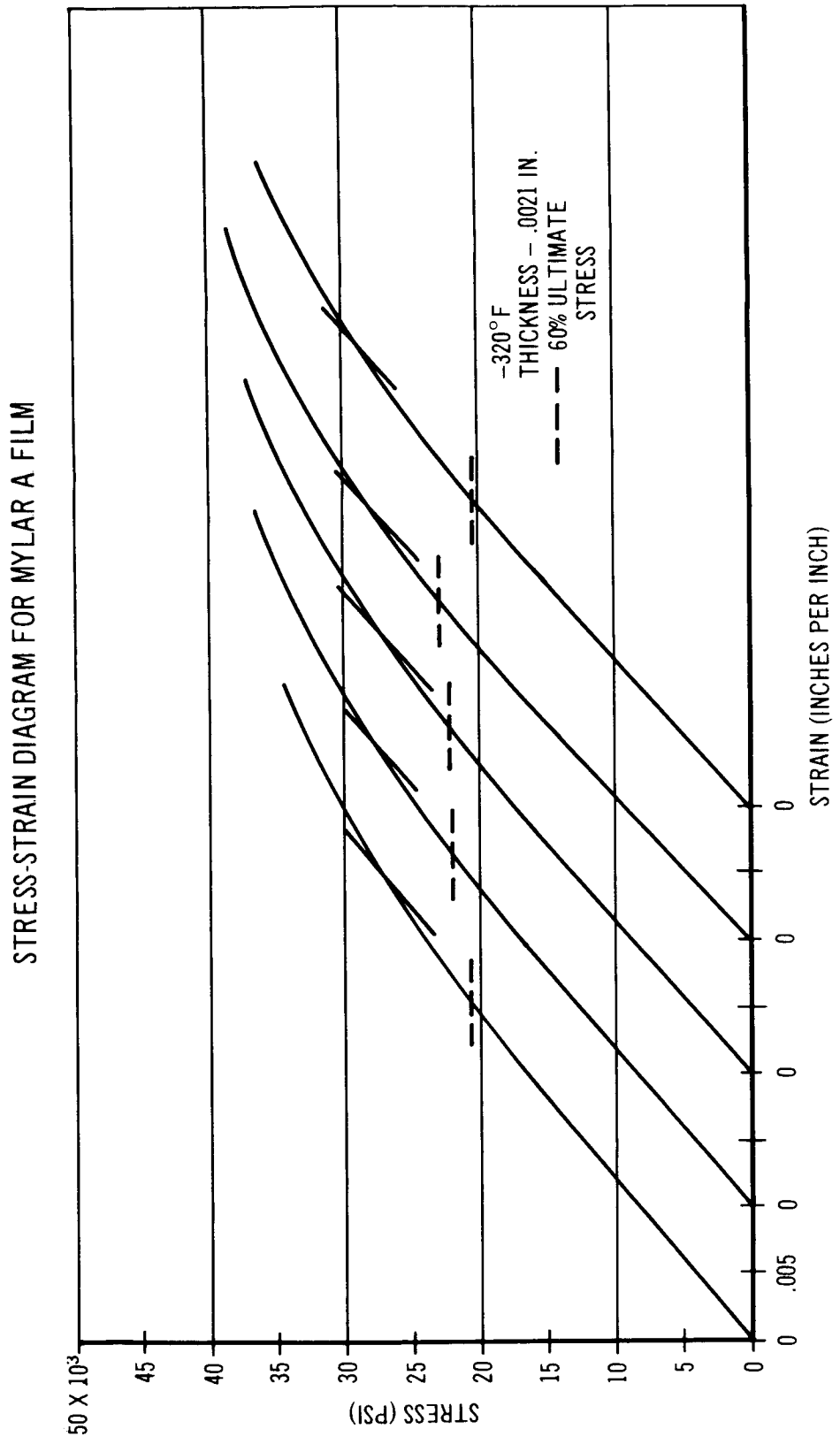

STRESS-STRAIN DIAGRAM FOR MYLAR A FILM
-320°F
THICKNESS – .0021 IN.
60% ULTIMATE STRESS
STRESS (PSI)
50 X 10³
45
40
35
30
25
20
15
10
5
0
0
.005
0
0
0
0
STRAIN (INCHES PER INCH)

FIGURE 2-5

STRESS-STRAIN DIAGRAM MYLAR A FILM

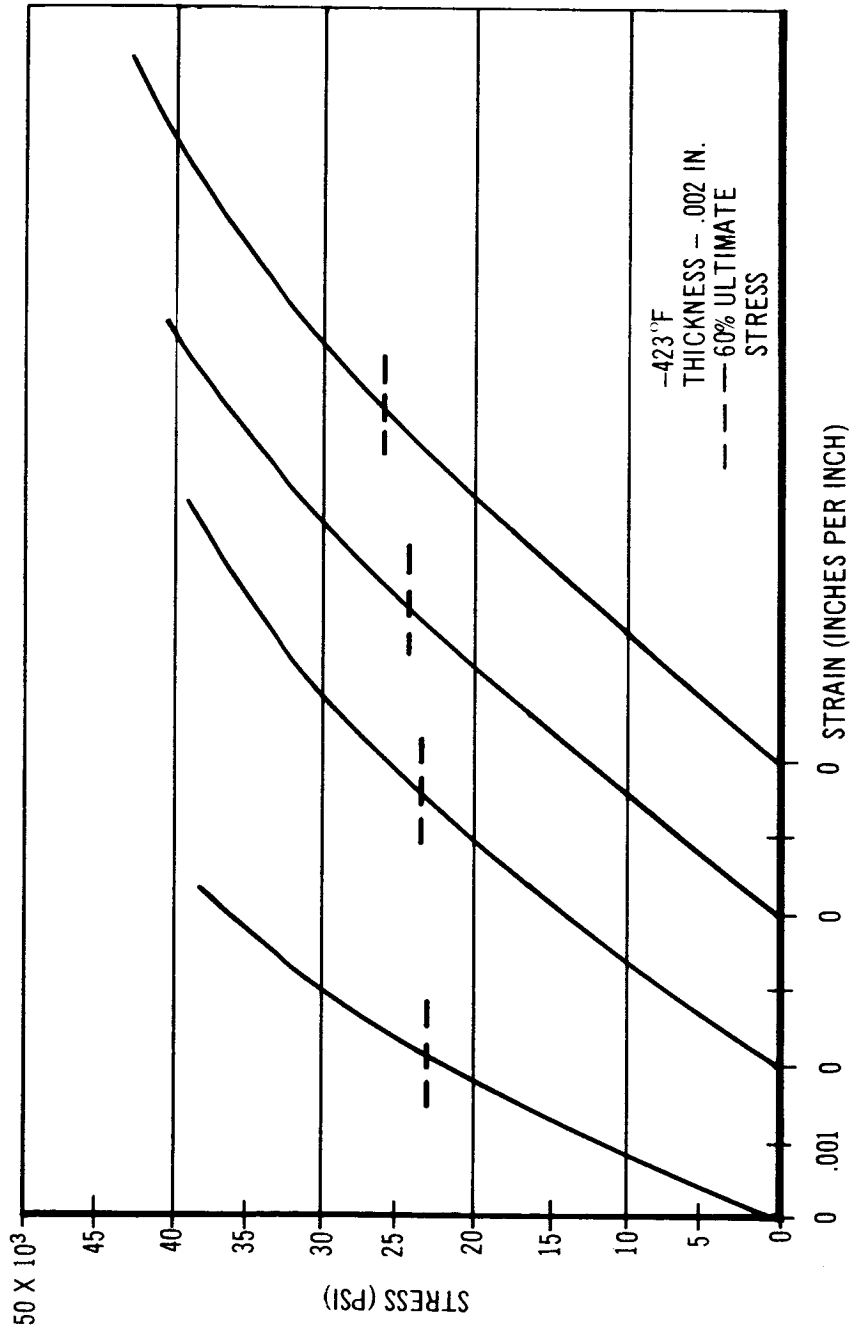


FIGURE 2-6

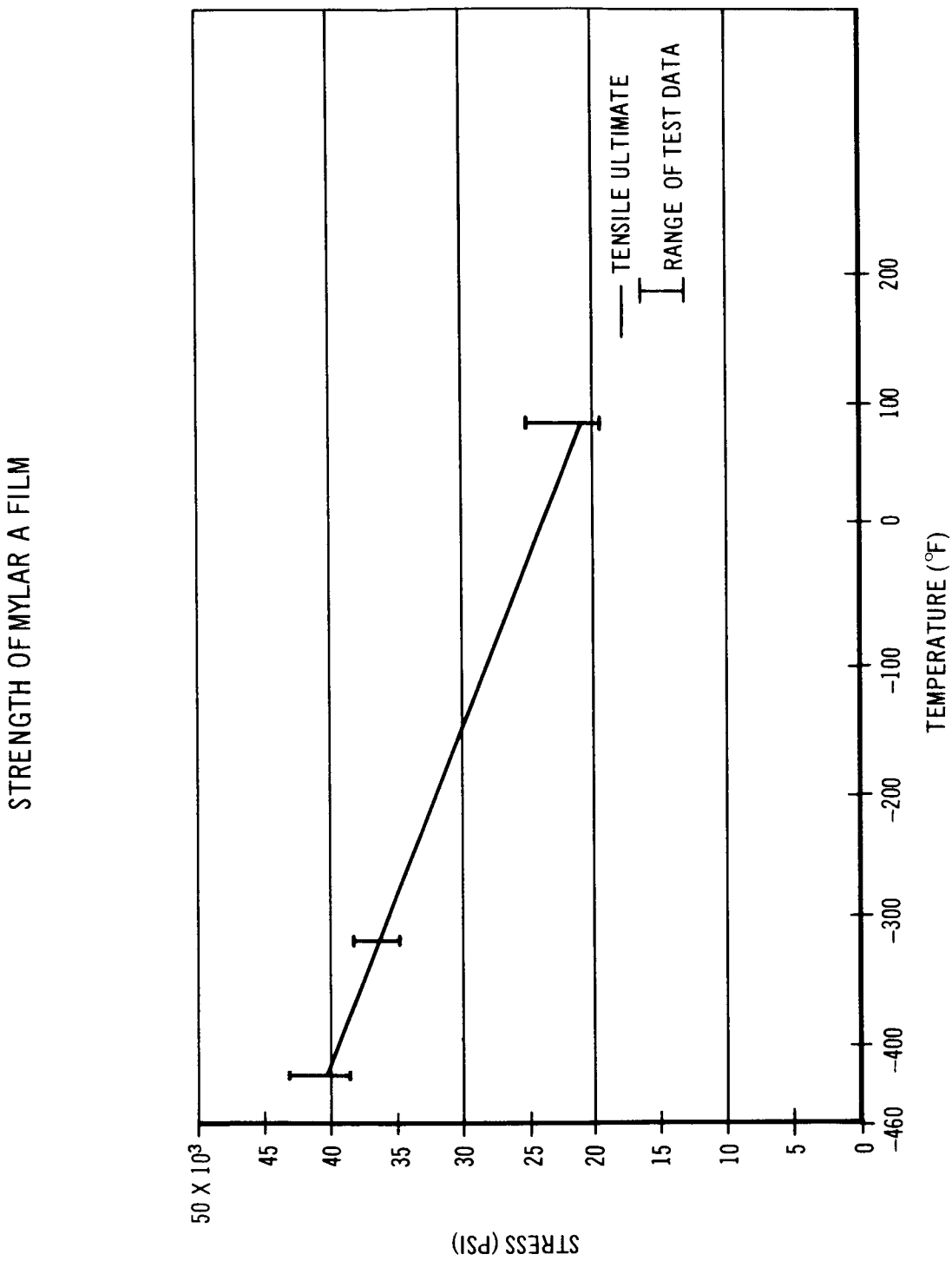

STRENGTH OF MYLAR A FILM
STRESS (PSI)
50 X 10³
45
40
35
30
25
20
15
10
5
0
TENSILE ULTIMATE
RANGE OF TEST DATA
200
100
0
-100
-200
-300
-400
-460
TEMPERATURE (°F)

FIGURE 2-7

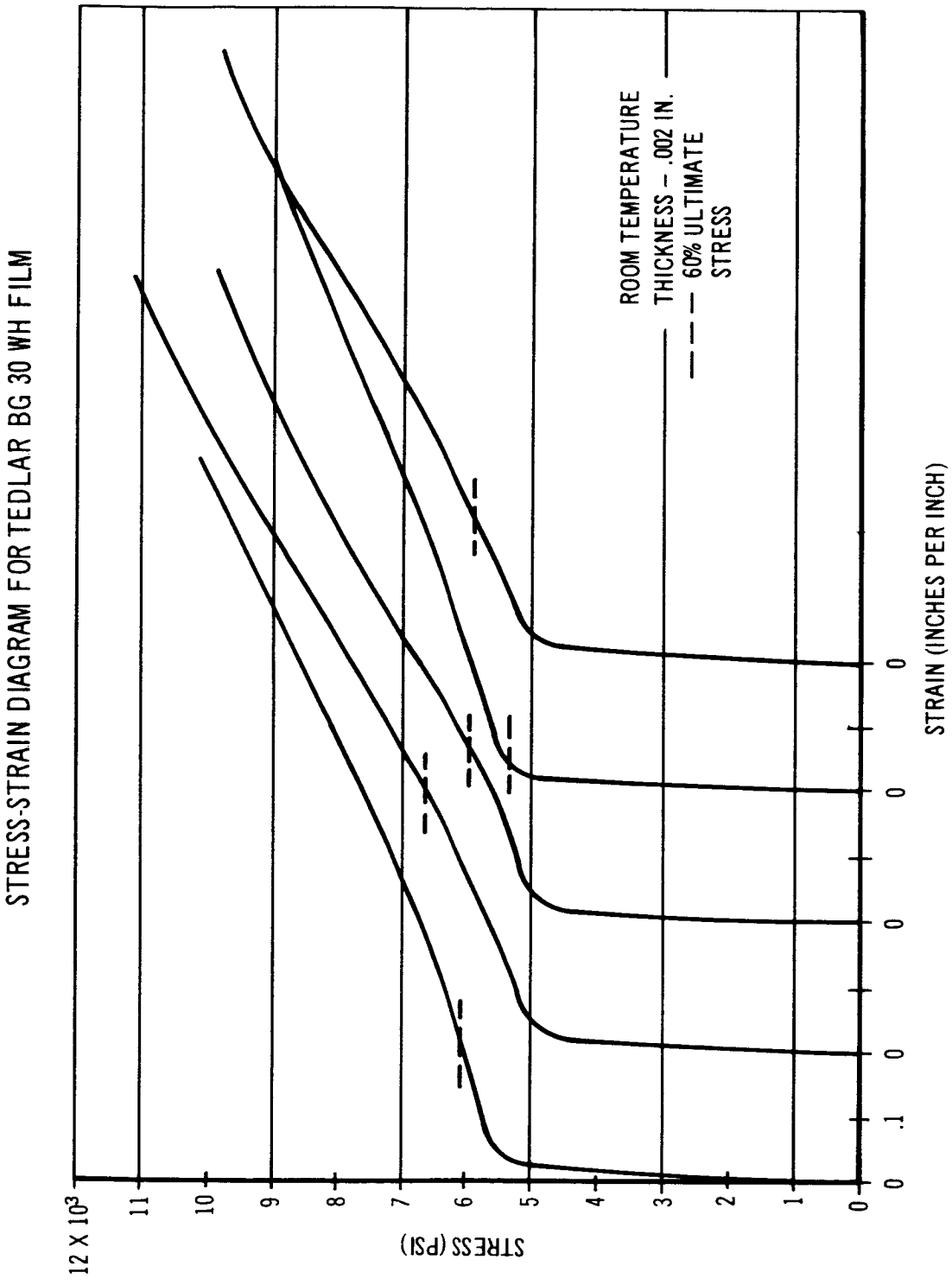

STRESS-STRAIN DIAGRAM FOR TEDLAR BG 30 WH FILM
ROOM TEMPERATURE
THICKNESS – .002 IN.
— — — 60% ULTIMATE STRESS
STRESS (PSI)
12 X 10³
11
10
9
8
7
6
5
4
3
2
1
0
0
.1
0
0
0
0
STRAIN (INCHES PER INCH)

FIGURE 2-8

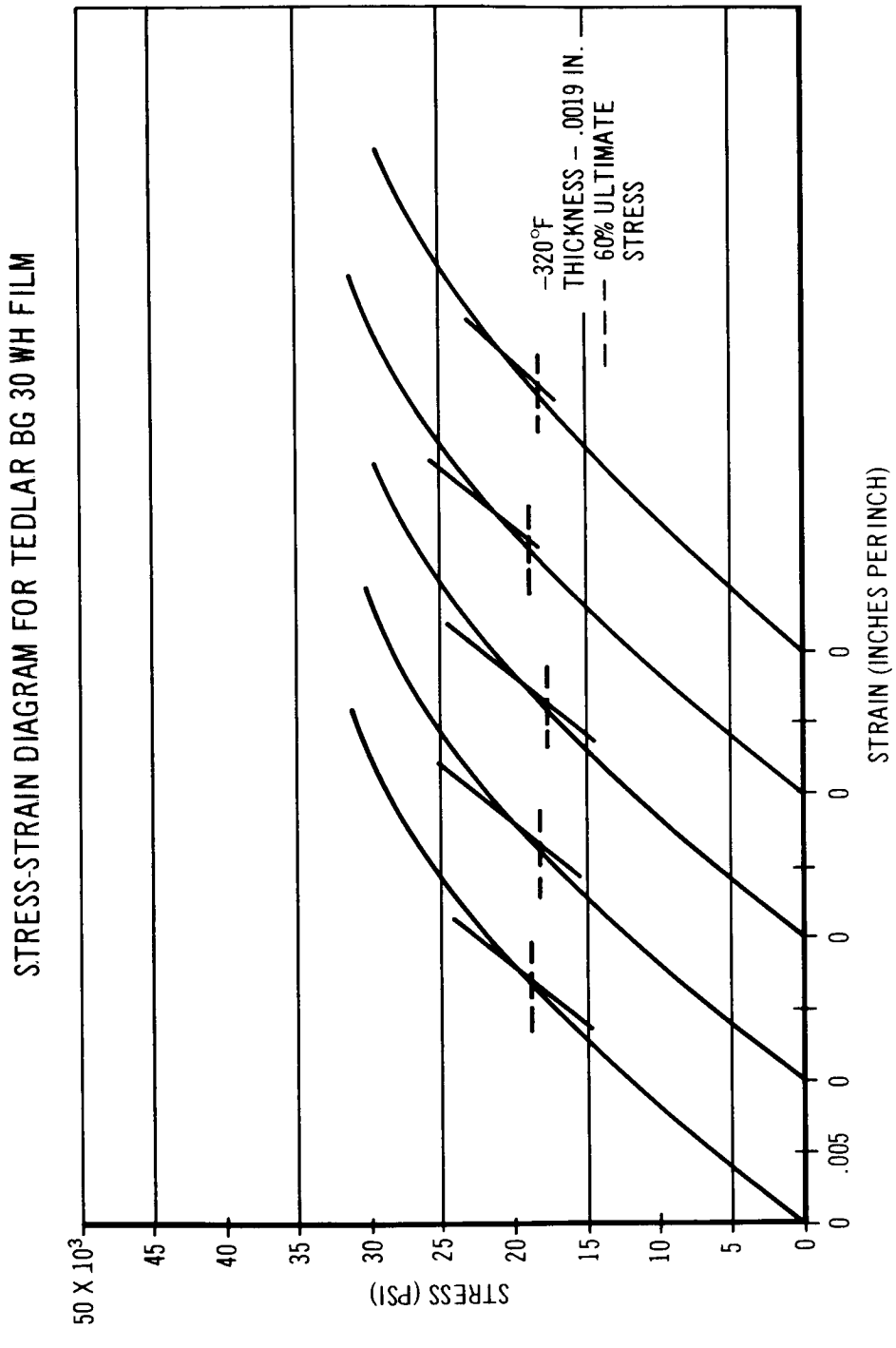

STRESS-STRAIN DIAGRAM FOR TEDLAR BG 30 WH FILM
50 X 10³
45
40
35
30
25
20
15
10
5
0
STRESS (PSI)
0
.005
0
0
0
0
STRAIN (INCHES PER INCH)
-320°F
THICKNESS – .0019 IN.
60% ULTIMATE
STRESS

FIGURE 2-9

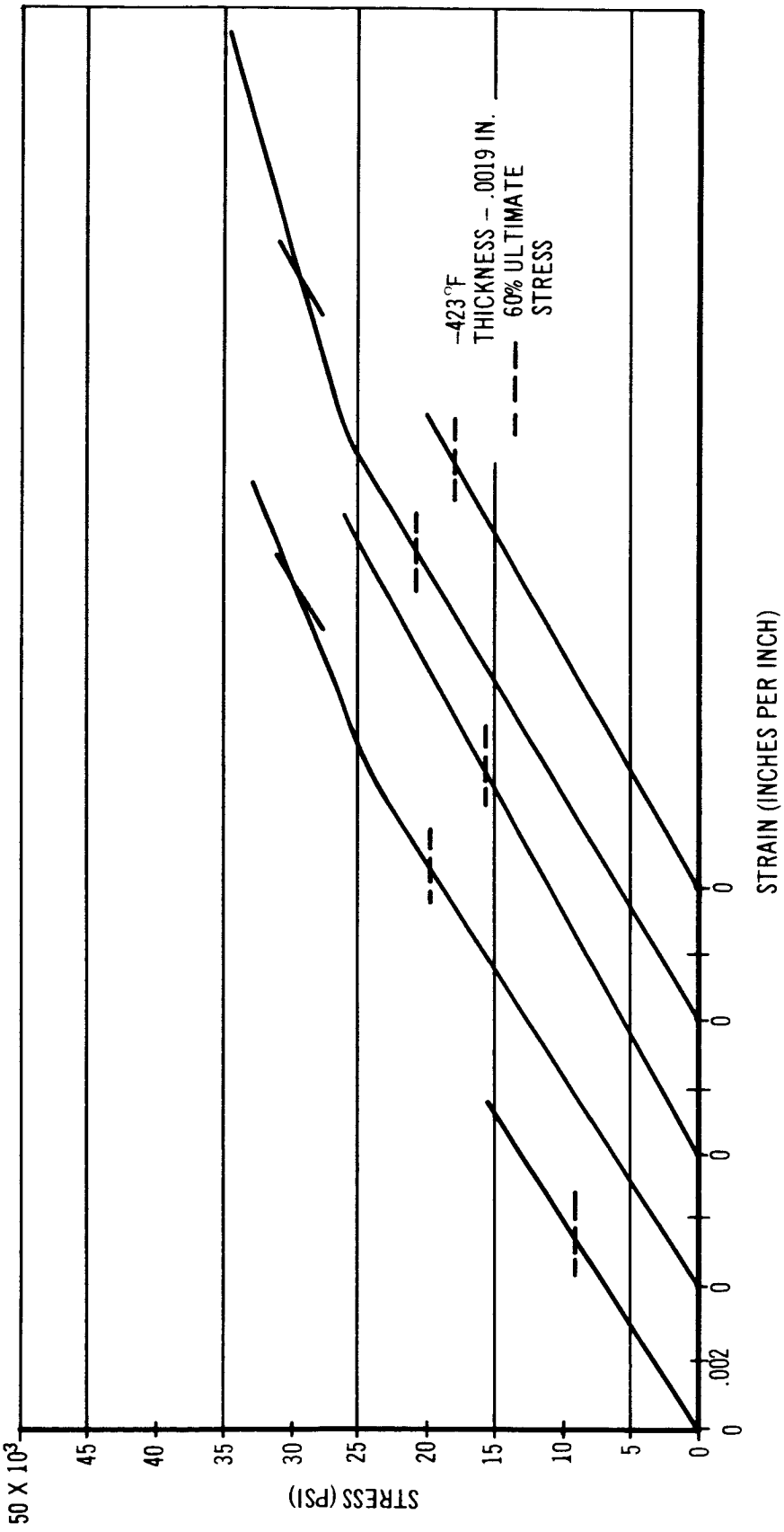

STRESS-STRAIN DIAGRAM FOR TEDLAR BG 30 WH FILM
-423°F
THICKNESS - .0019 IN.
60% ULTIMATE STRESS
STRESS (PSI)
50 X 10³
45
40
35
30
25
20
15
10
5
0
STRAIN (INCHES PER INCH)
0
.002
0
0
0
0
0

FIGURE 2-10

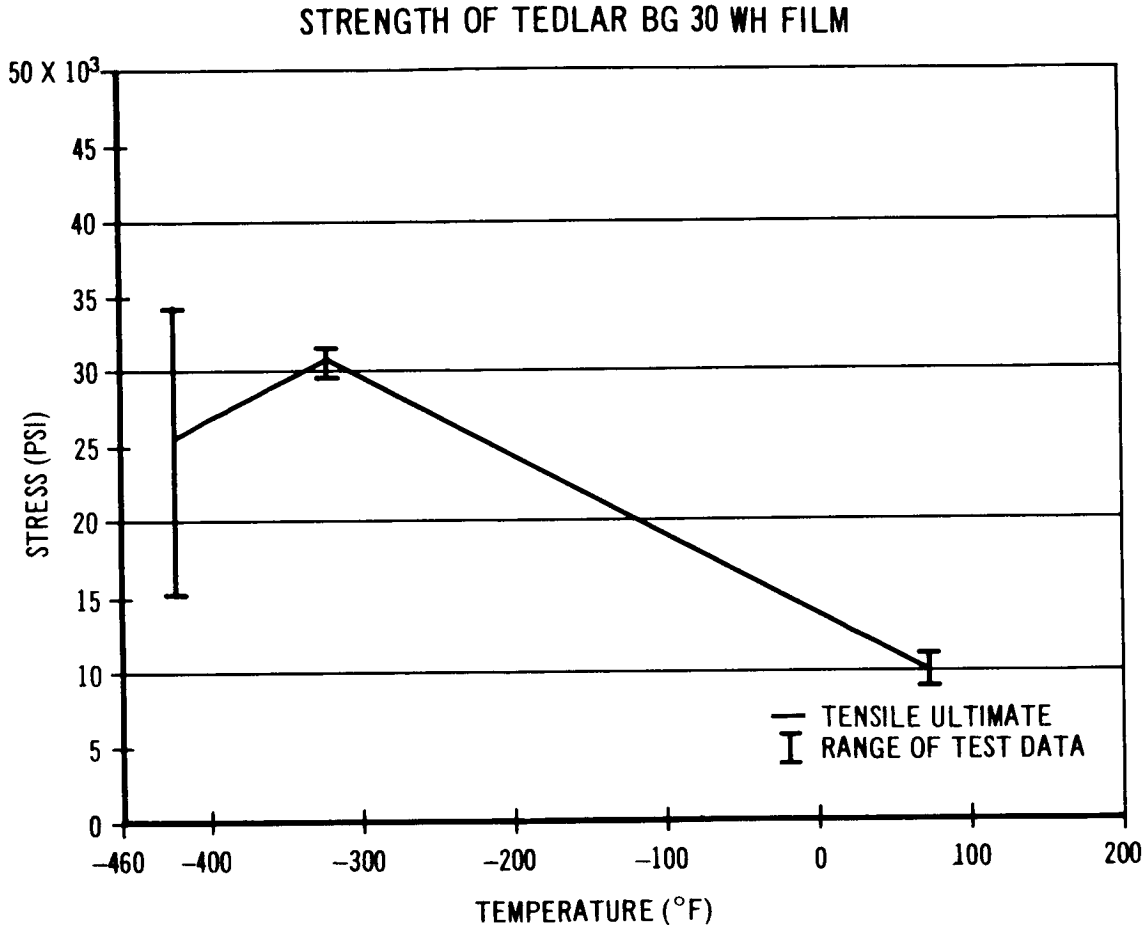

STRENGTH OF TEDLAR BG 30 WH FILM
50 X 10³
45
40
35
30
25
20
15
10
5
0
STRESS (PSI)
TENSILE ULTIMATE
RANGE OF TEST DATA
-460
-400
-300
-200
-100
0
100
200
TEMPERATURE (°F)

FIGURE 2-11

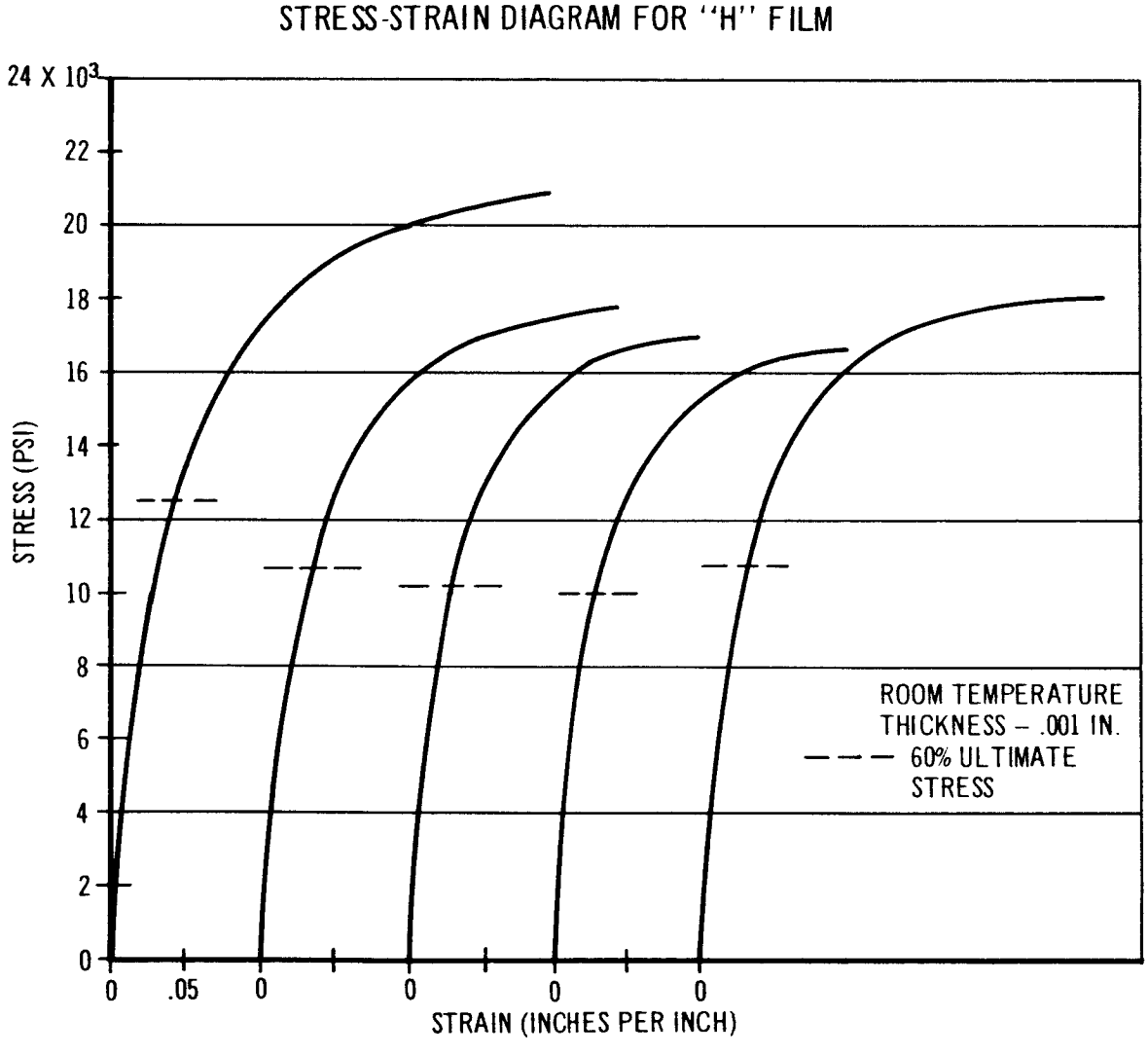

STRESS-STRAIN DIAGRAM FOR "H" FILM
24 X 10³
22
20
18
16
14
12
10
8
6
4
2
0
STRESS (PSI)
0
.05
0
0
0
0
STRAIN (INCHES PER INCH)
ROOM TEMPERATURE
THICKNESS – .001 IN.
60% ULTIMATE STRESS

FIGURE 2-12

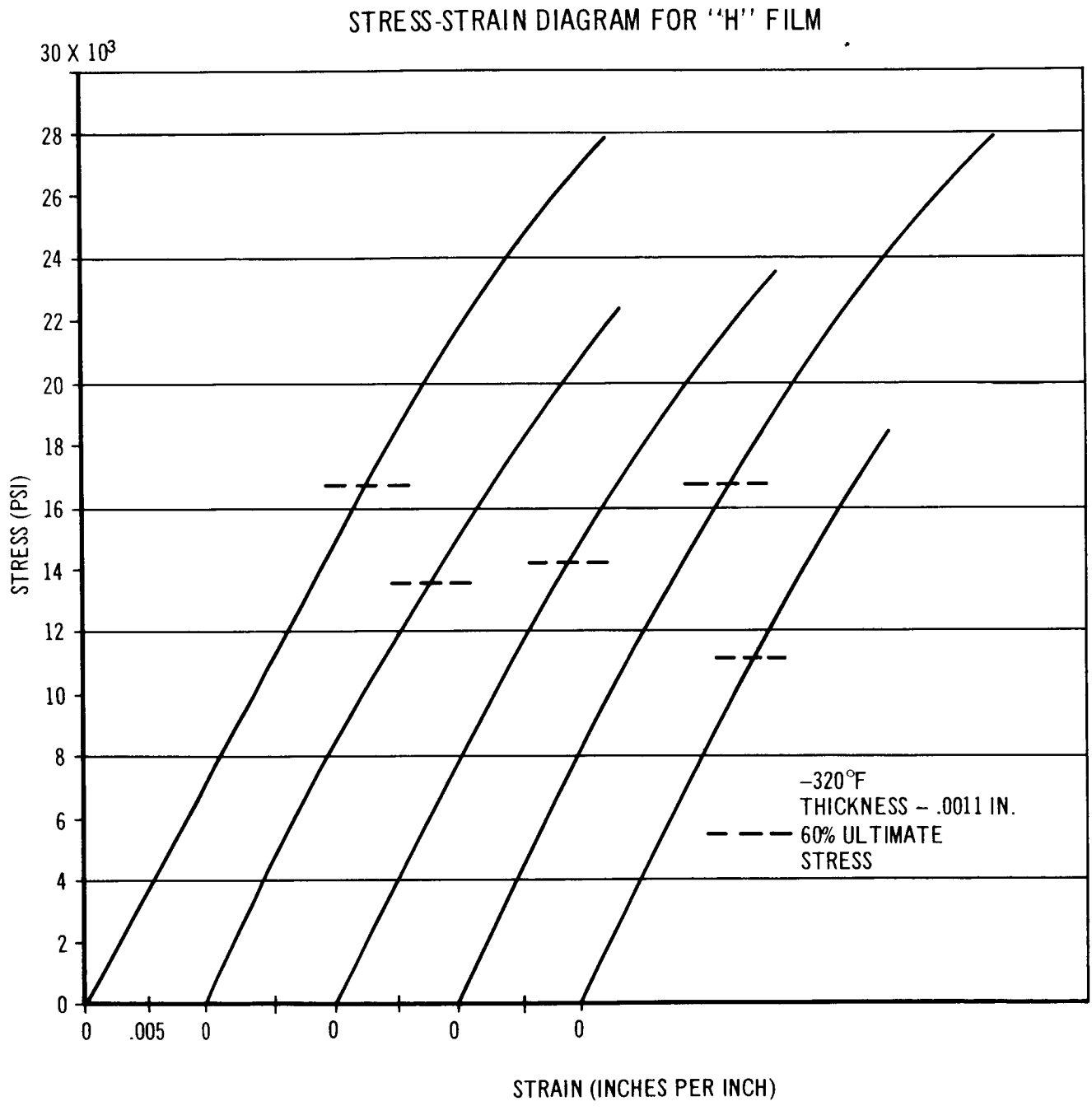

STRESS-STRAIN DIAGRAM FOR "H" FILM
30 X 10³
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
STRESS (PSI)
0
.005
0
0
0
0
STRAIN (INCHES PER INCH)
–320°F
THICKNESS – .0011 IN.
60% ULTIMATE
STRESS

FIGURE 2-13

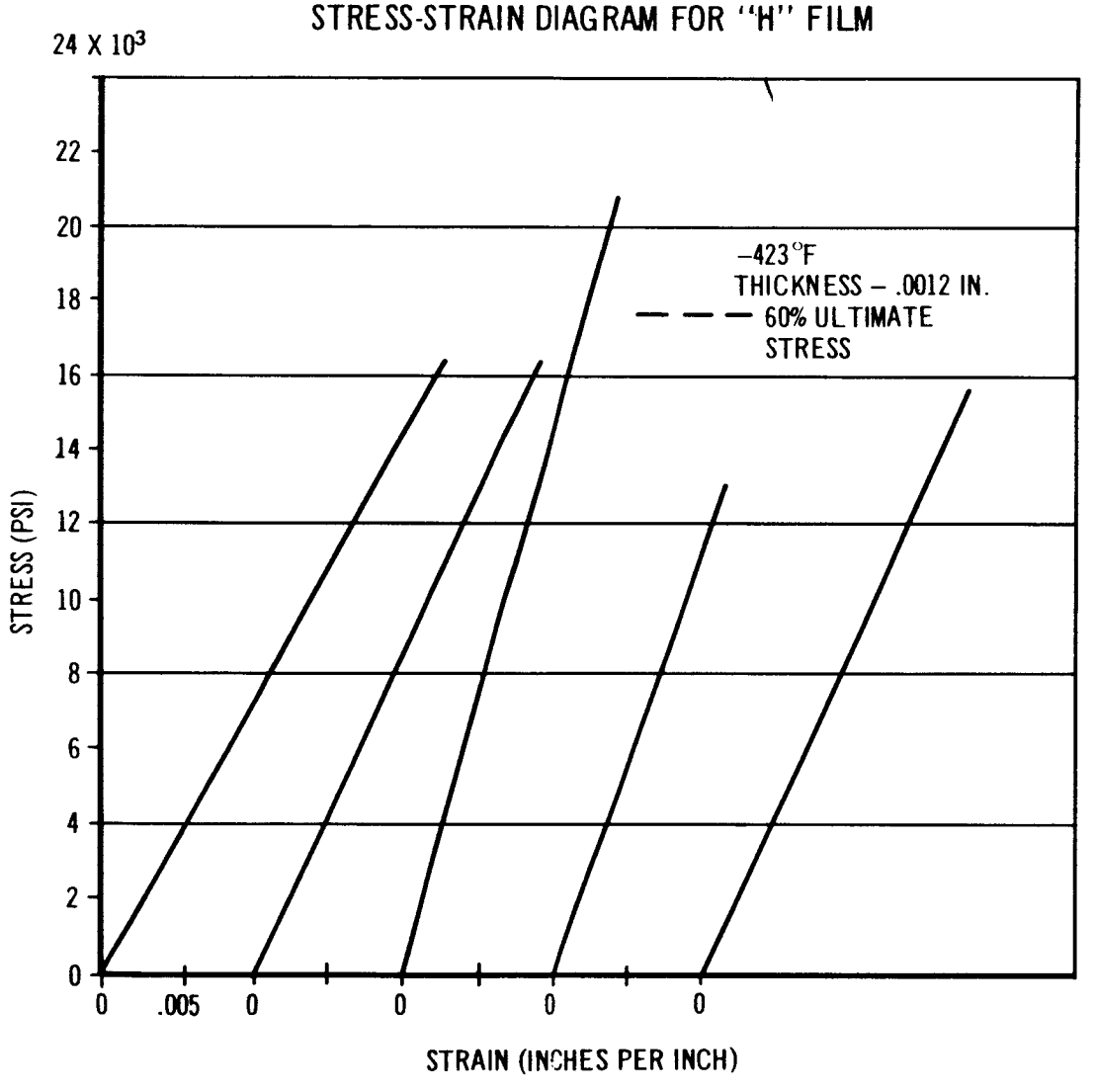

STRESS-STRAIN DIAGRAM FOR "H" FILM
24 X 10³
22
20
18
16
14
12
10
8
6
4
2
0
STRESS (PSI)
−423°F
THICKNESS – .0012 IN.
60% ULTIMATE
STRESS
0
.005
0
0
0
0
STRAIN (INCHES PER INCH)

FIGURE 2-14

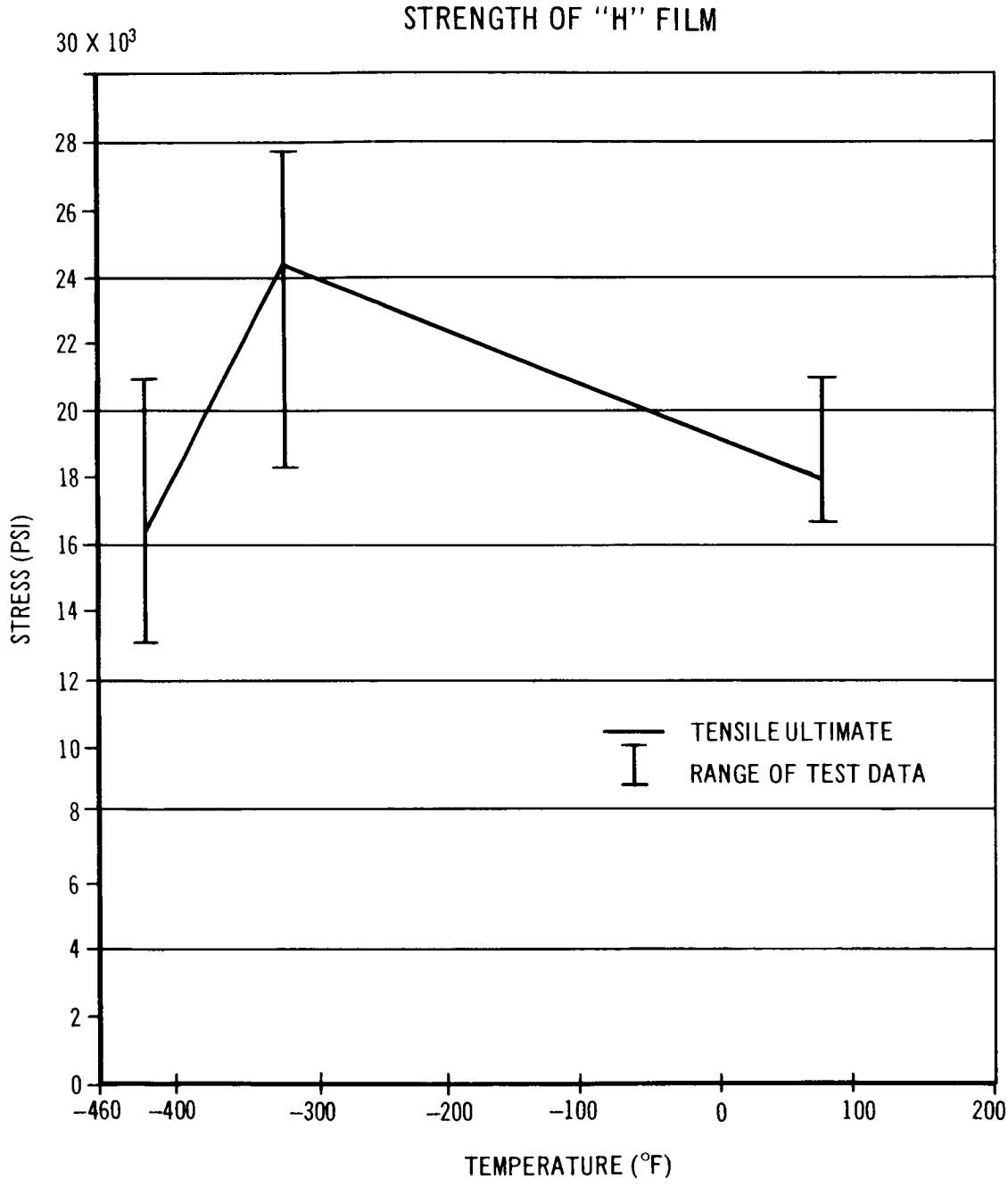

STRENGTH OF "H" FILM
30 X 10³
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
STRESS (PSI)
TENSILE ULTIMATE
RANGE OF TEST DATA
-460
-400
-300
-200
-100
0
100
200
TEMPERATURE (°F)

FIGURE 2-15

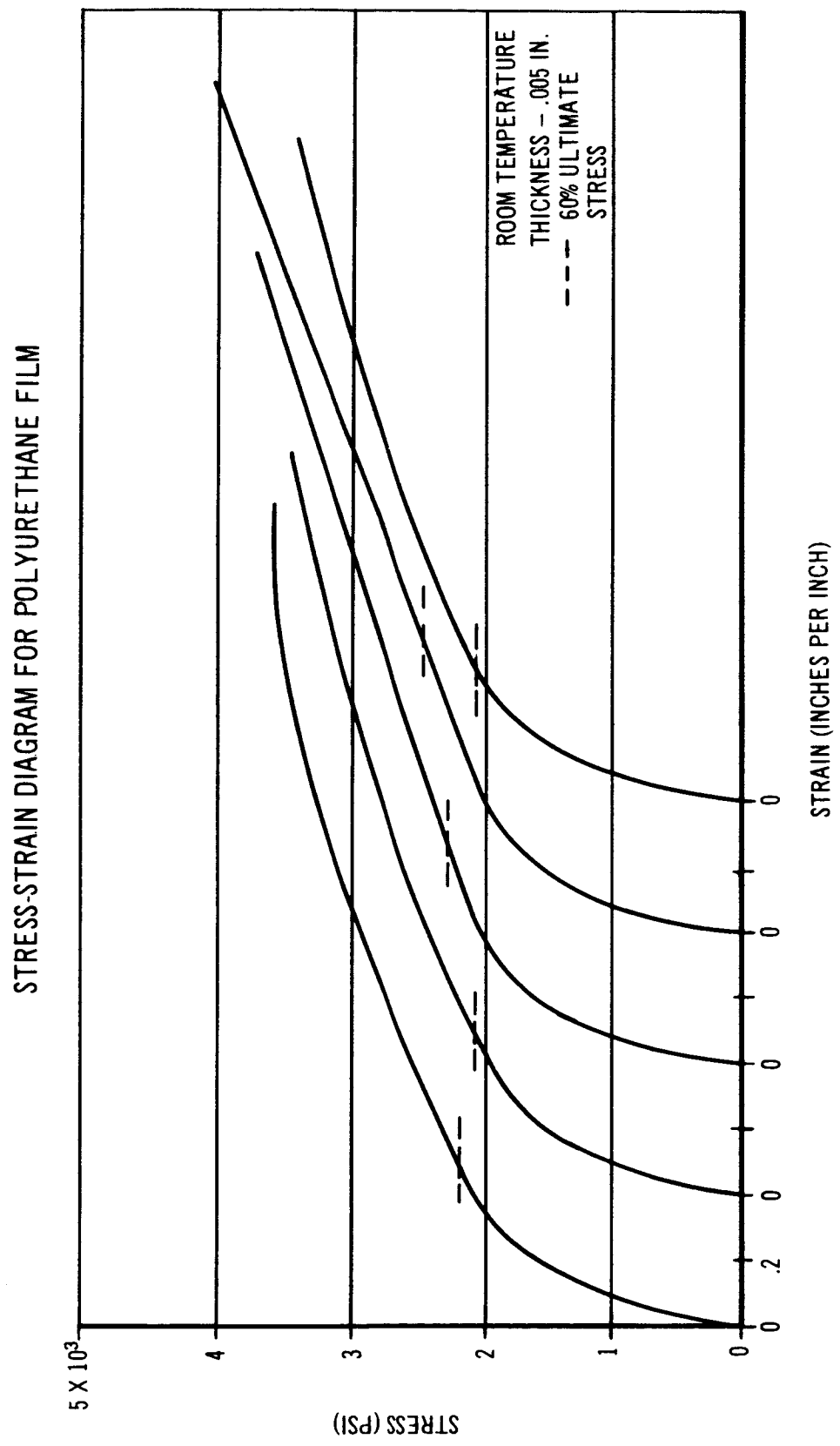

STRESS-STRAIN DIAGRAM FOR POLYURETHANE FILM
ROOM TEMPERATURE
THICKNESS - .005 IN.
60% ULTIMATE STRESS
STRESS (PSI)
5 X 10³
4
3
2
1
0
STRAIN (INCHES PER INCH)
0
.2
0
0
0
0

FIGURE 2-16

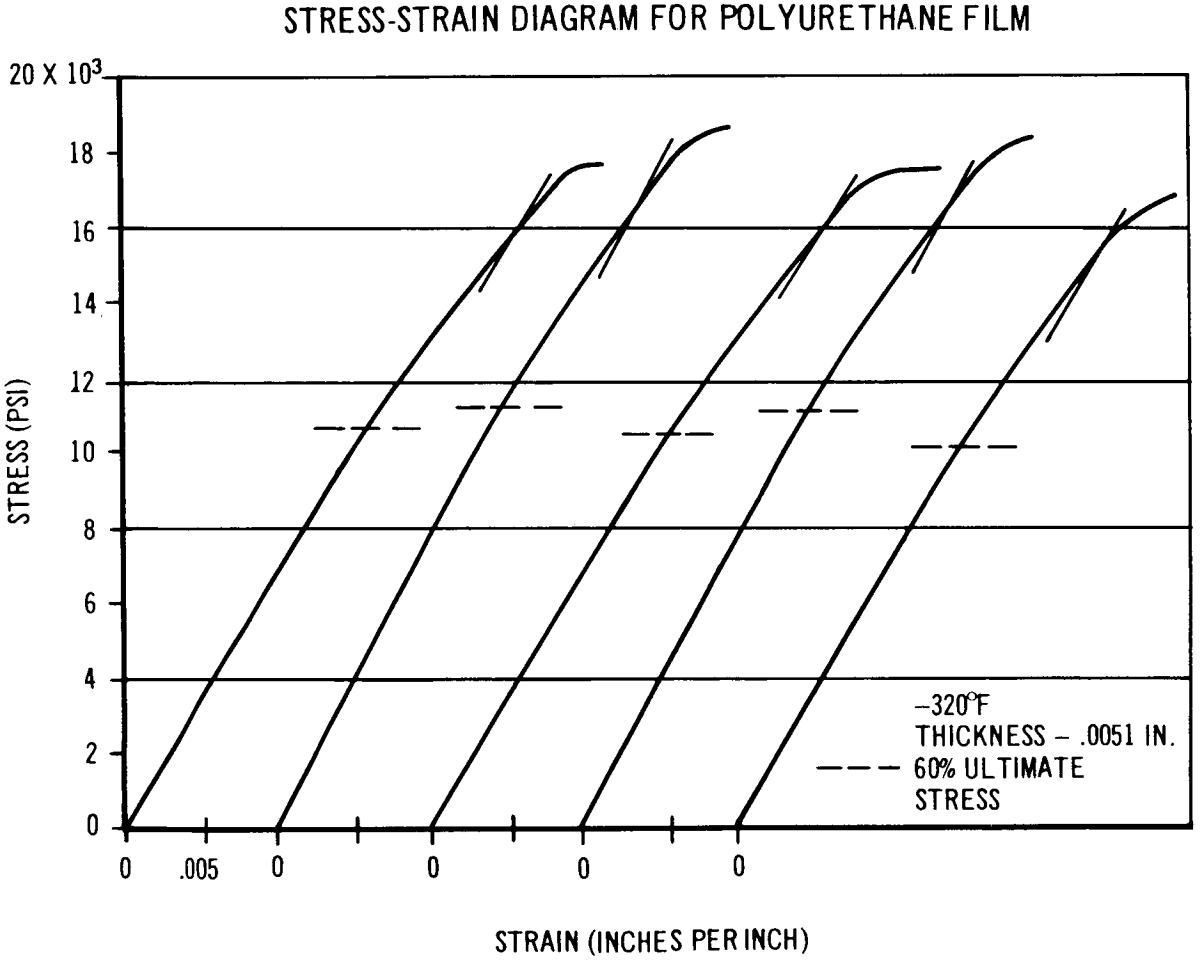

STRESS-STRAIN DIAGRAM FOR POLYURETHANE FILM
20 X 10³
18
16
14
12
10
8
6
4
2
0
STRESS (PSI)
0
.005
0
0
0
0
STRAIN (INCHES PER INCH)
–320°F
THICKNESS – .0051 IN.
60% ULTIMATE
STRESS

FIGURE 2-17

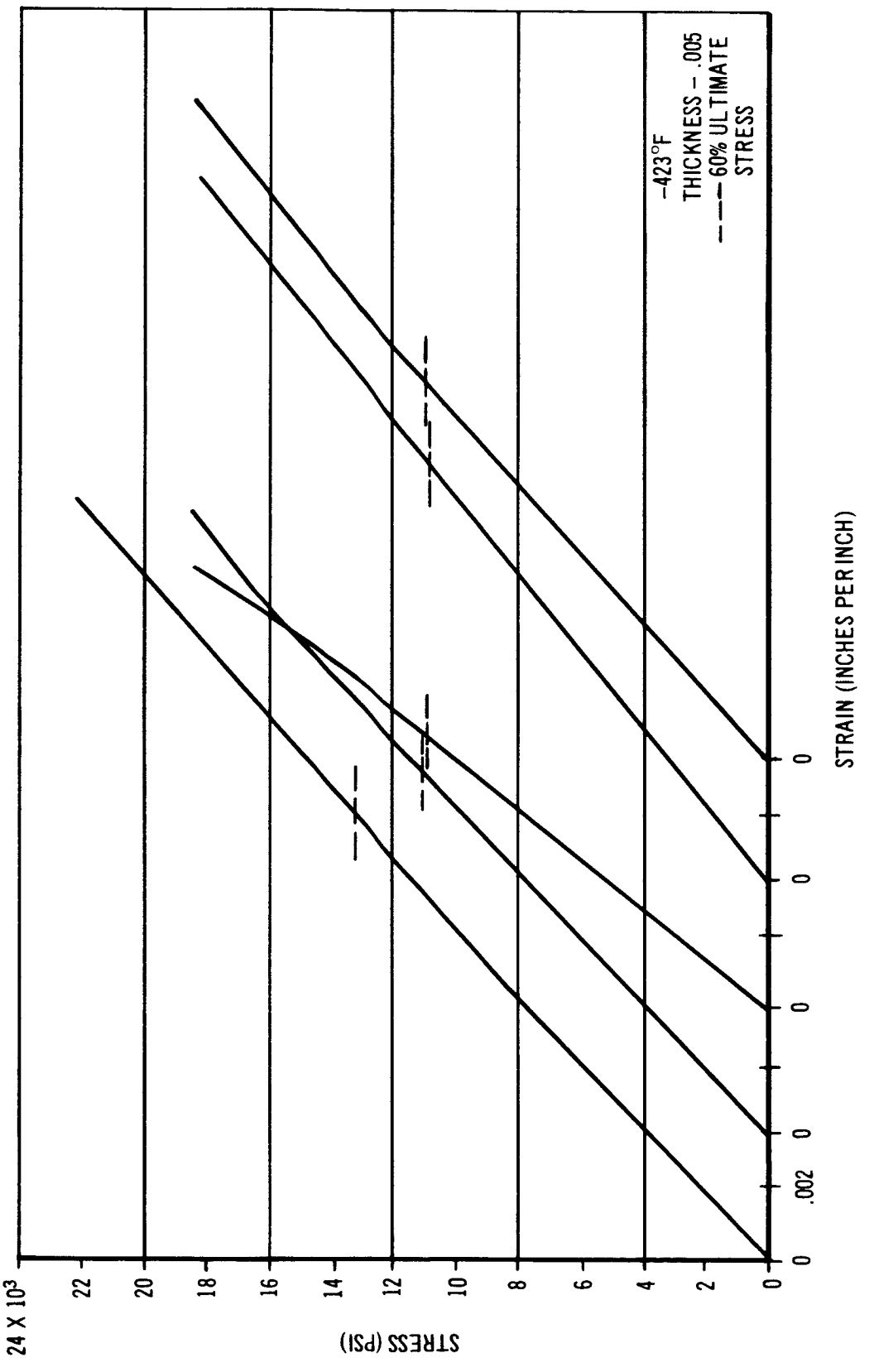

STRESS-STRAIN DIAGRAM FOR POLYURETHANE FILM
-423°F
THICKNESS – .005
60% ULTIMATE STRESS
STRESS (PSI)
24 X 10³
22
20
18
16
14
12
10
8
6
4
2
0
0
.002
0
0
0
0
STRAIN (INCHES PER INCH)

FIGURE 2-18

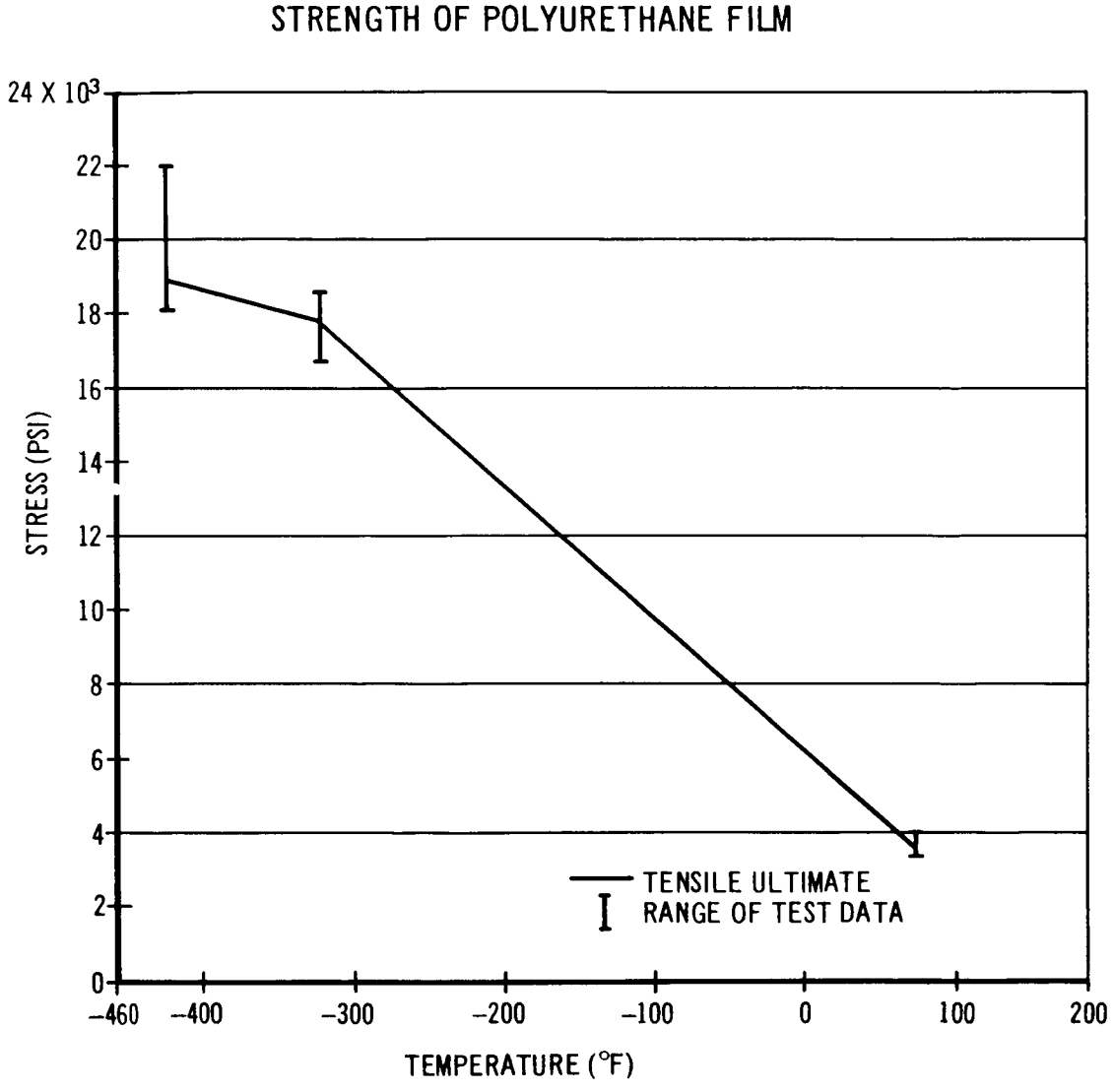

STRENGTH OF POLYURETHANE FILM
24 X 10³
22
20
18
16
14
12
10
8
6
4
2
0
STRESS (PSI)
TENSILE ULTIMATE
RANGE OF TEST DATA
-460
-400
-300
-200
-100
0
100
200
TEMPERATURE (°F)

FIGURE 2-19

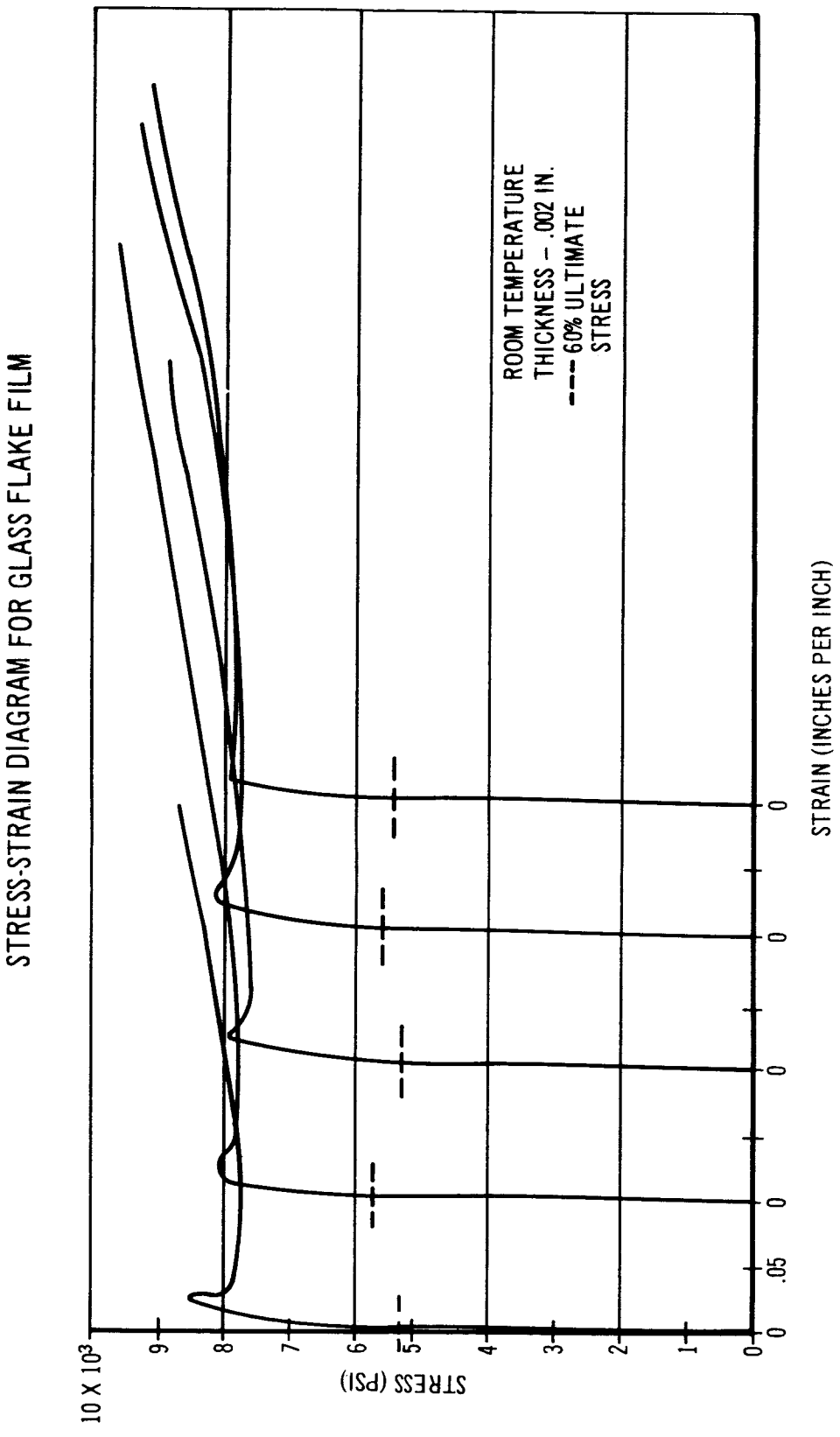

STRESS-STRAIN DIAGRAM FOR GLASS FLAKE FILM
ROOM TEMPERATURE
THICKNESS – .002 IN.
– – – 60% ULTIMATE STRESS
STRESS (PSI)
10 X 10³
9
8
7
6
5
4
3
2
1
0
0
.05
0
0
0
0
STRAIN (INCHES PER INCH)

FIGURE 2-20

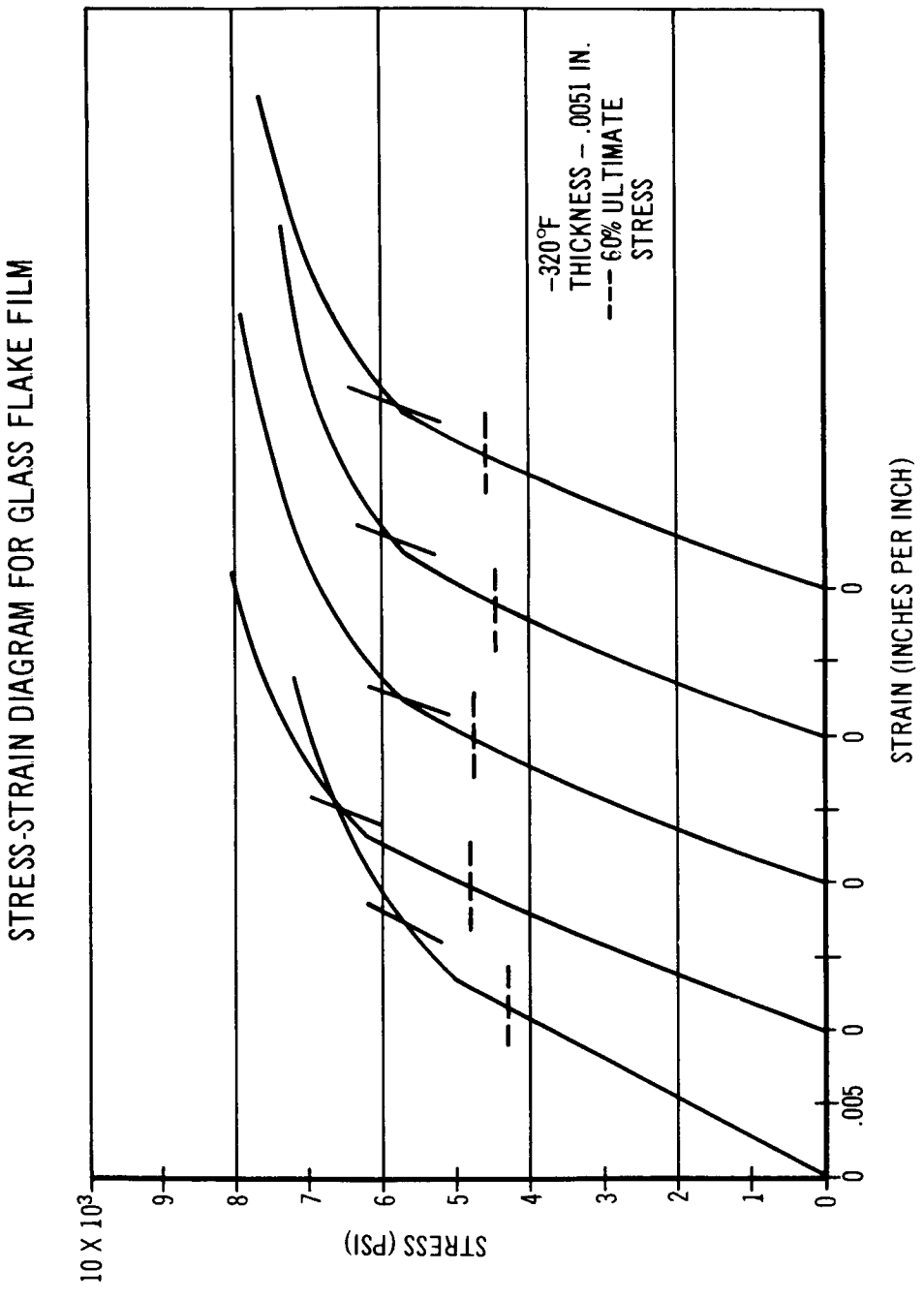

STRESS-STRAIN DIAGRAM FOR GLASS FLAKE FILM
-320°F
THICKNESS - .0051 IN.
--- 60% ULTIMATE STRESS
10 X 10³
STRESS (PSI)
9
8
7
6
5
4
3
2
1
0
0
.005
0
0
0
0
STRAIN (INCHES PER INCH)

FIGURE 2-21

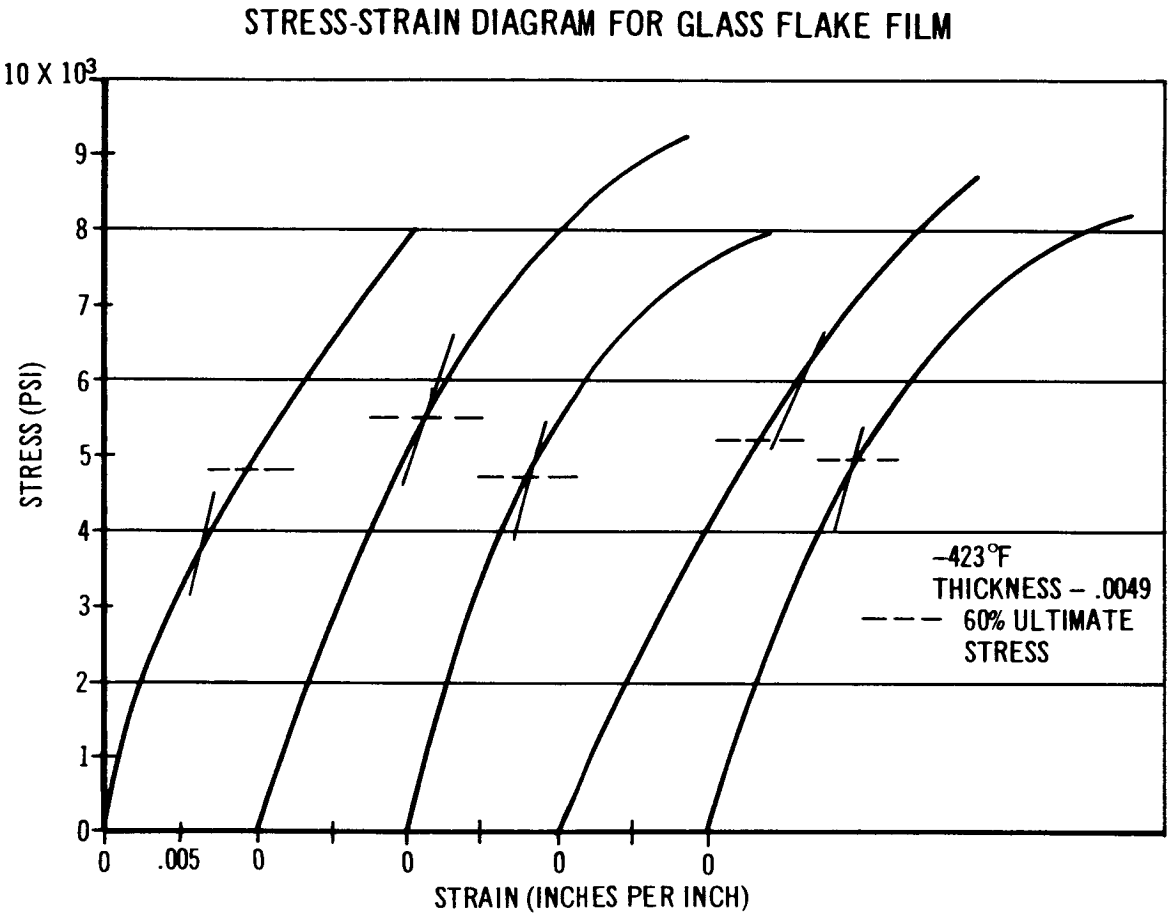

STRESS-STRAIN DIAGRAM FOR GLASS FLAKE FILM
10 X 10³
9
8
7
6
5
4
3
2
1
0
STRESS (PSI)
0
.005
0
0
0
0
STRAIN (INCHES PER INCH)
-423°F
THICKNESS - .0049
60% ULTIMATE STRESS

FIGURE 2-22

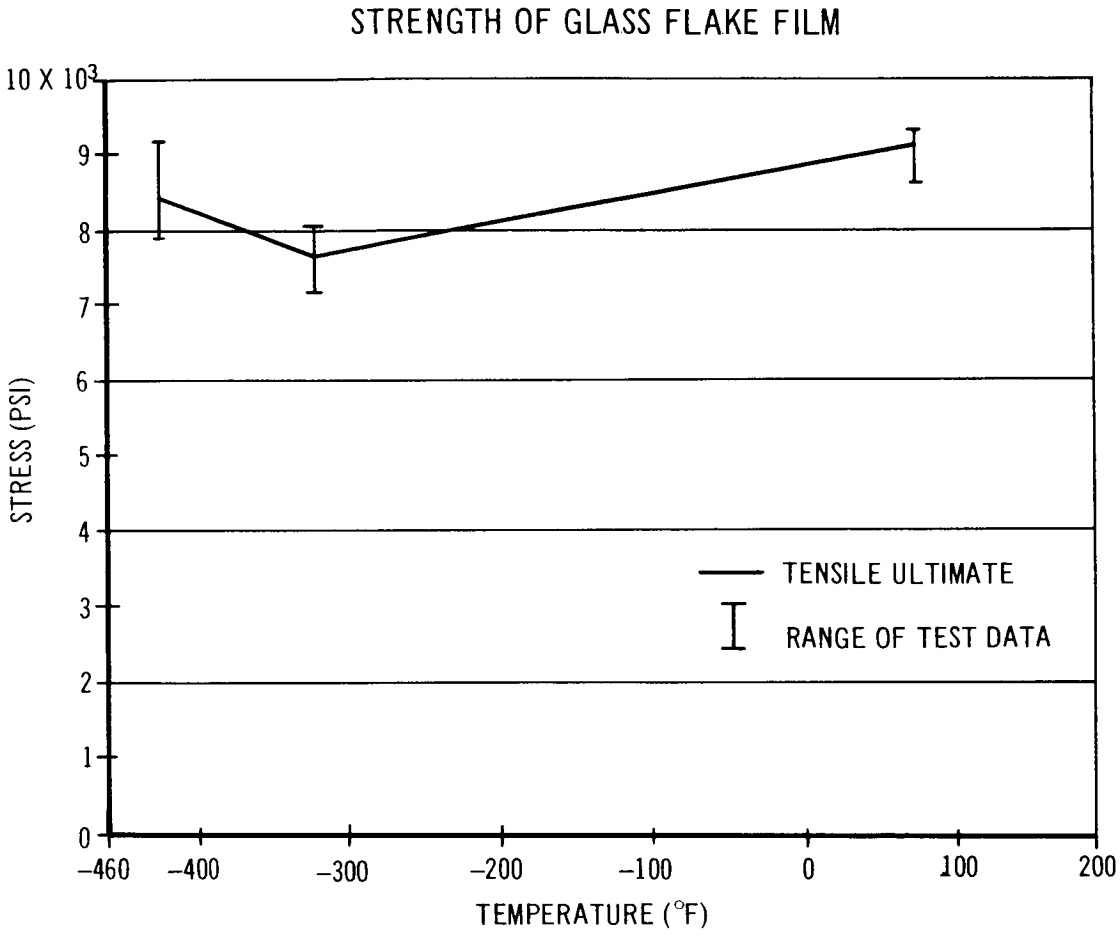

STRENGTH OF GLASS FLAKE FILM
10 X 10³
9
8
7
6
5
4
3
2
1
0
STRESS (PSI)
TENSILE ULTIMATE
RANGE OF TEST DATA
-460
-400
-300
-200
-100
0
100
200
TEMPERATURE (°F)

FIGURE 2-23

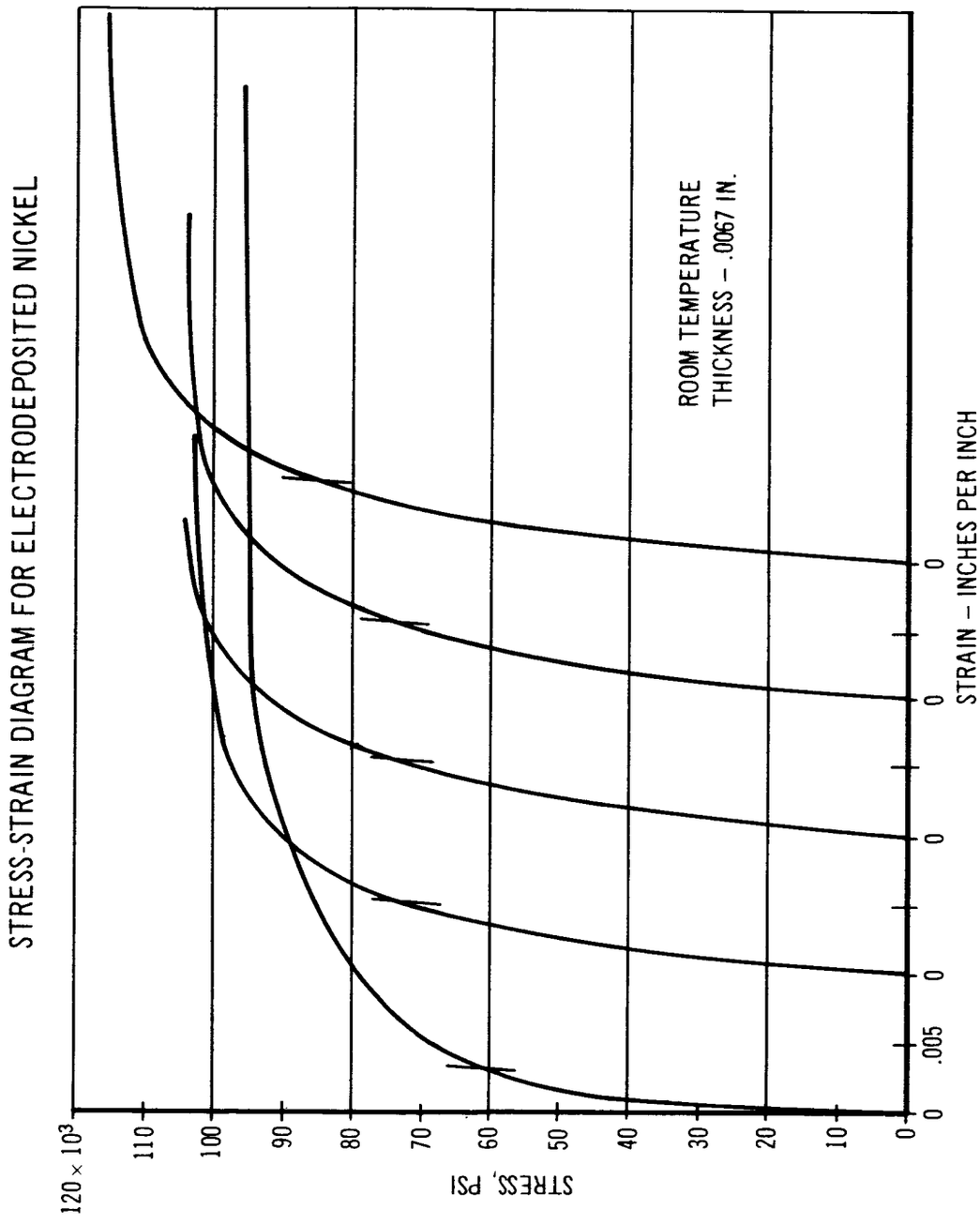

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED NICKEL
ROOM TEMPERATURE
THICKNESS – .0067 IN.
STRESS, PSI
120 × 10³
110
100
90
80
70
60
50
40
30
20
10
0
0
.005
0
0
0
0
0
STRAIN – INCHES PER INCH

FIGURE 2-24

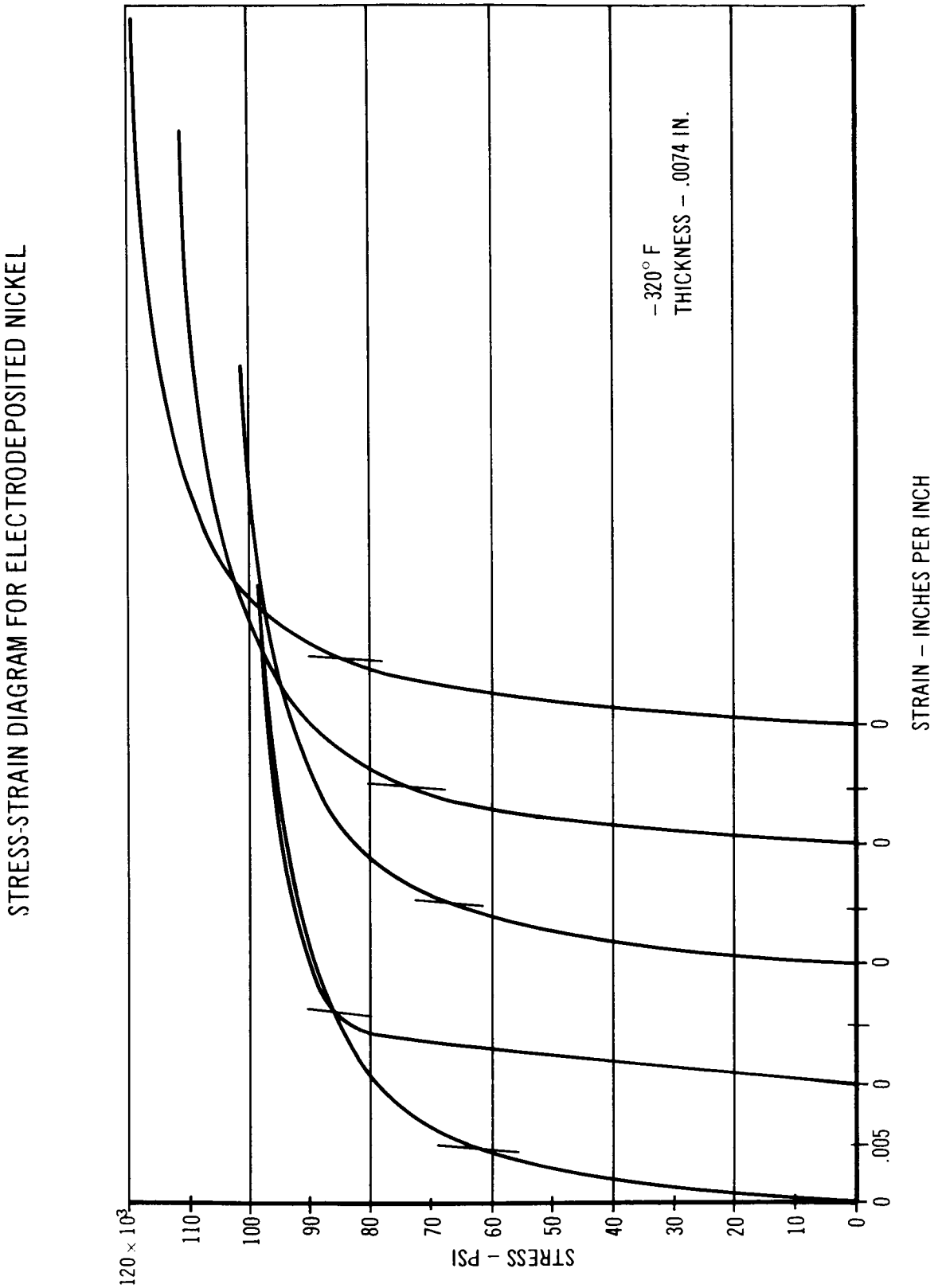

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED NICKEL
-320° F
THICKNESS – .0074 IN.
STRESS – PSI
120 × 10³
110
100
90
80
70
60
50
40
30
20
10
0
0
0
0
0
0
.005
STRAIN – INCHES PER INCH

FIGURE 2-25

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED NICKEL

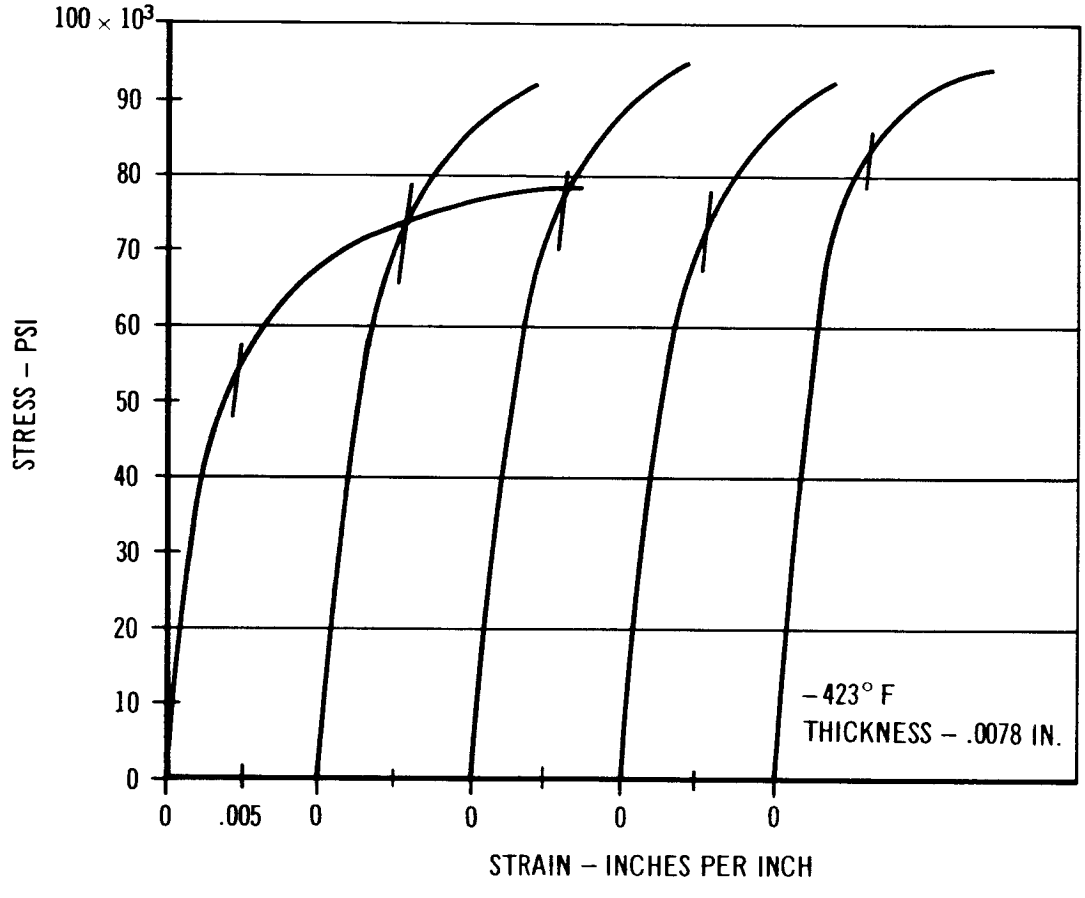


FIGURE 2-26

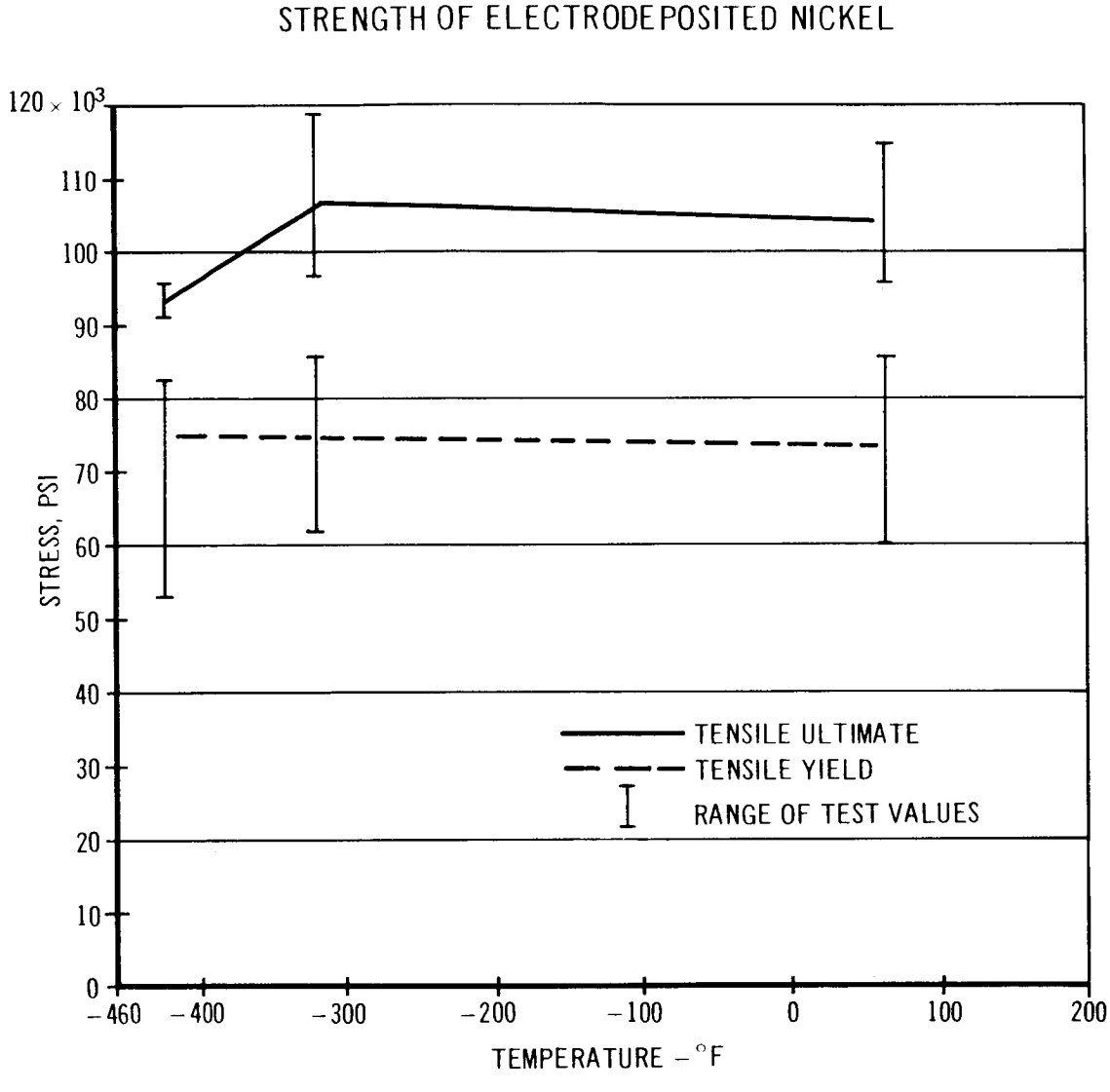

STRENGTH OF ELECTRODEPOSITED NICKEL
120 × 10³
110
100
90
80
70
60
50
40
30
20
10
0
STRESS, PSI
TENSILE ULTIMATE
TENSILE YIELD
RANGE OF TEST VALUES
−460
−400
−300
−200
−100
0
100
200
TEMPERATURE − °F

FIGURE 2-27

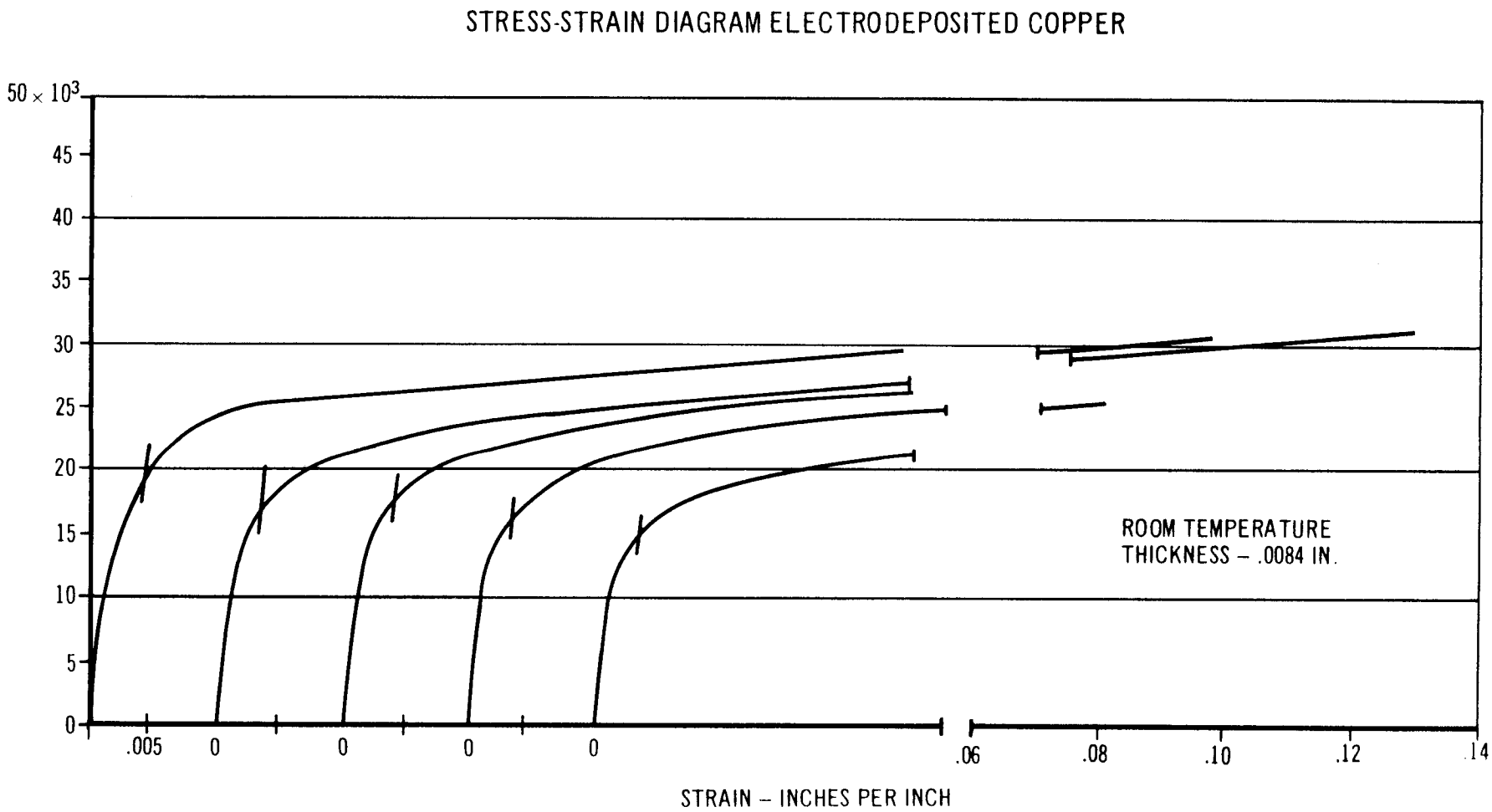

STRESS-STRAIN DIAGRAM ELECTRODEPOSITED COPPER
50 × 10³
45
40
35
30
25
20
15
10
5
0
STRESS – PSI
.005
0
0
0
0
.06
.08
.10
.12
.14
STRAIN – INCHES PER INCH
ROOM TEMPERATURE
THICKNESS – .0084 IN.

FIGURE 2-28

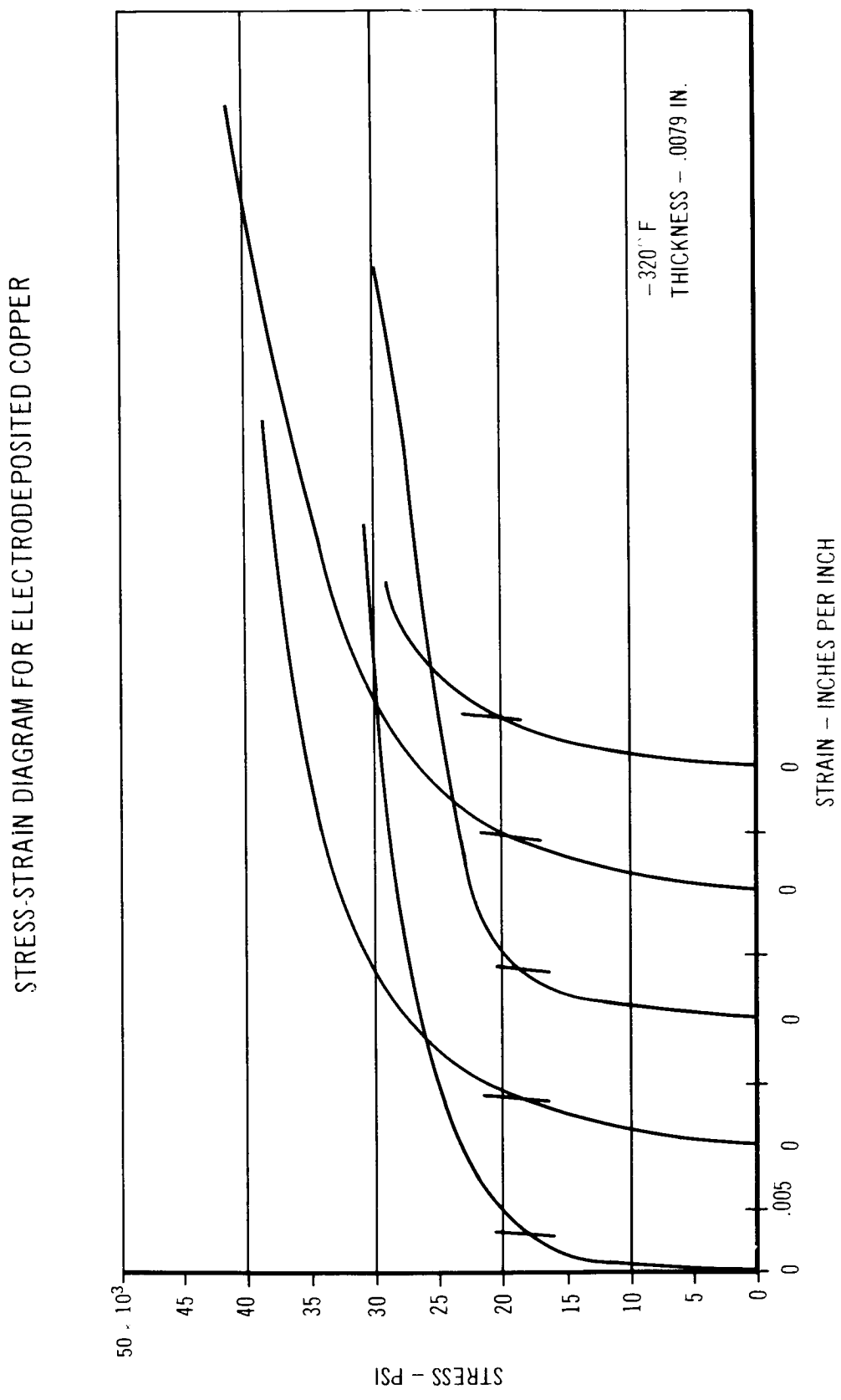

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED COPPER
-320° F
THICKNESS – .0079 IN.
STRESS – PSI
50 · 10³
45
40
35
30
25
20
15
10
5
0
STRAIN – INCHES PER INCH
0
.005
0
0
0
0

FIGURE 2-29

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED COPPER

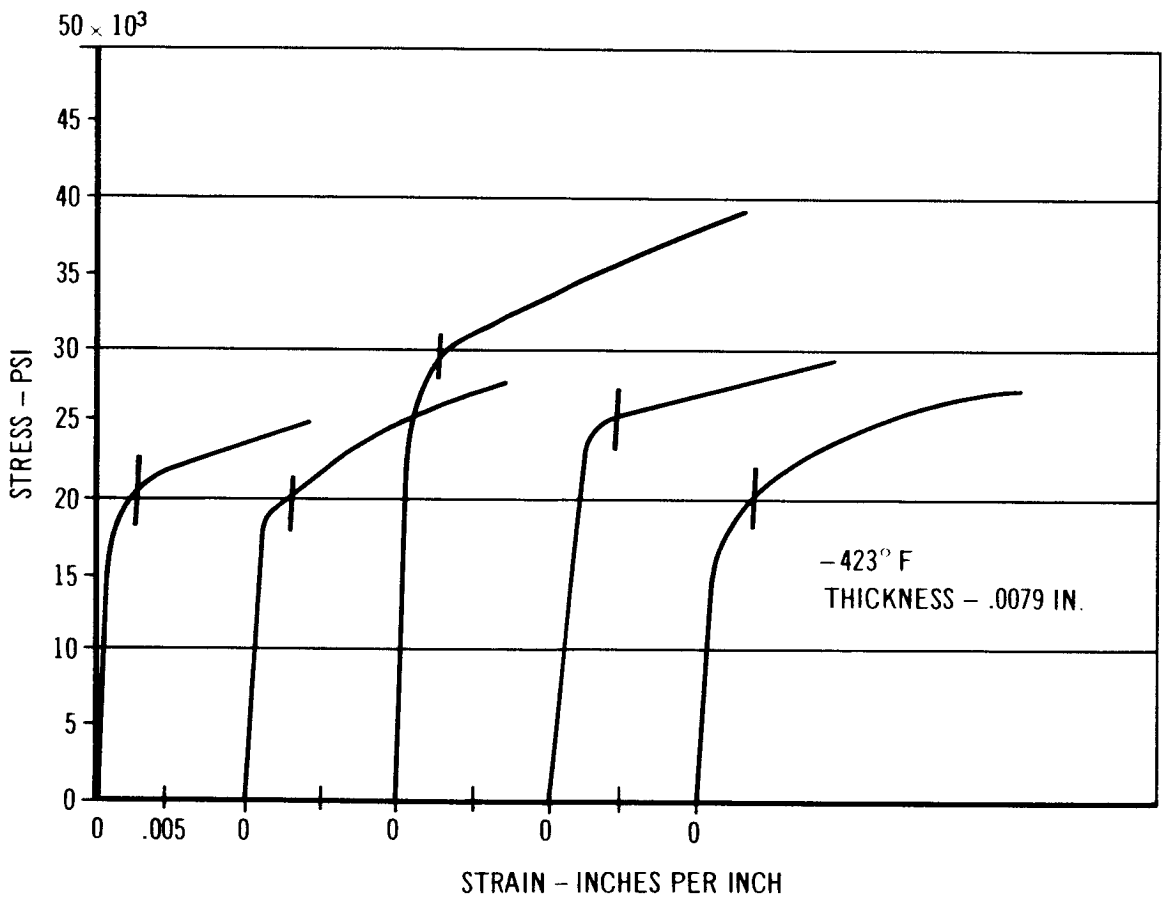


FIGURE 2-30

STRENGTH OF ELECTRODEPOSITED COPPER

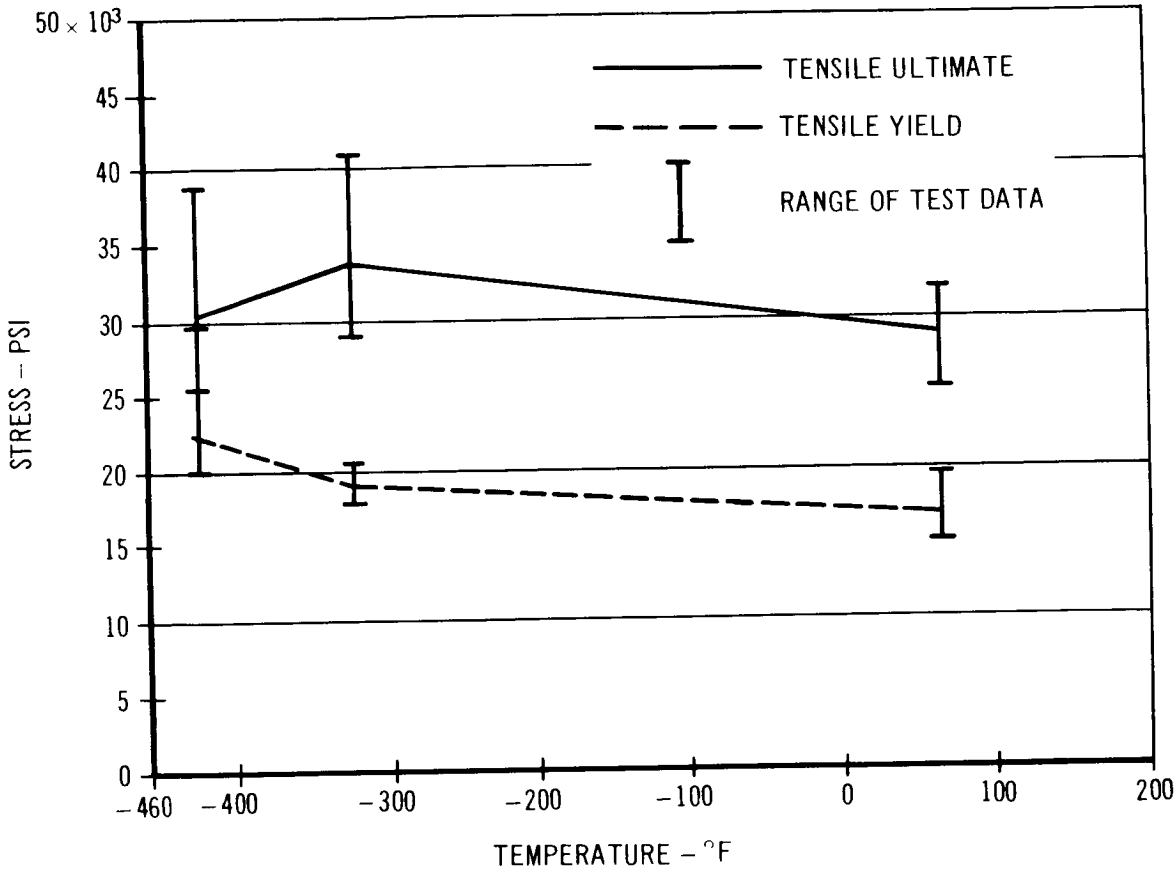


FIGURE 2-31

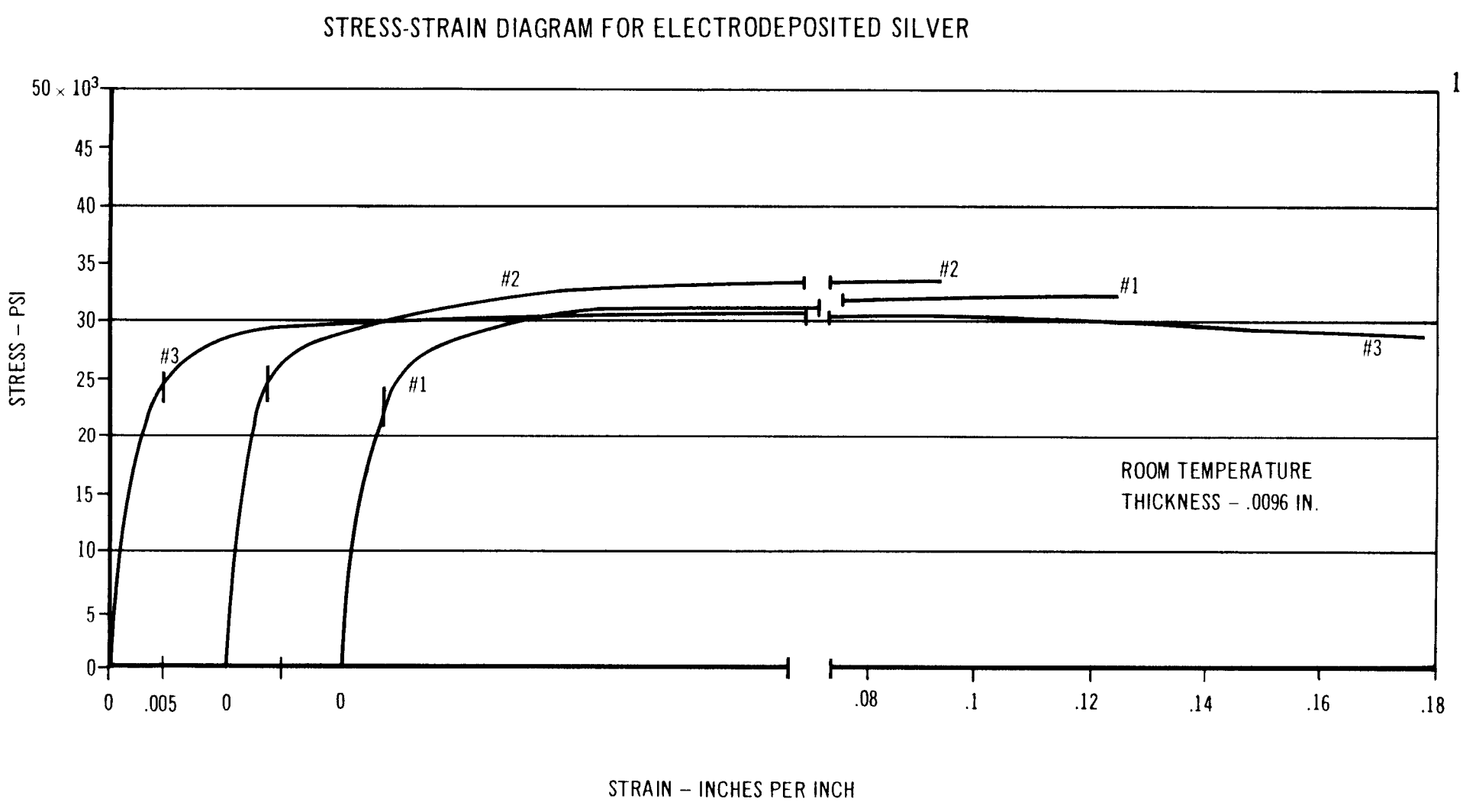

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED SILVER
50 × 10³
45
40
35
30
25
20
15
10
5
0
STRESS – PSI
#2
#2
#1
#3
#3
#1
ROOM TEMPERATURE
THICKNESS – .0096 IN.
0
.005
0
0
.08
.1
.12
.14
.16
.18
STRAIN – INCHES PER INCH

FIGURE 2-32

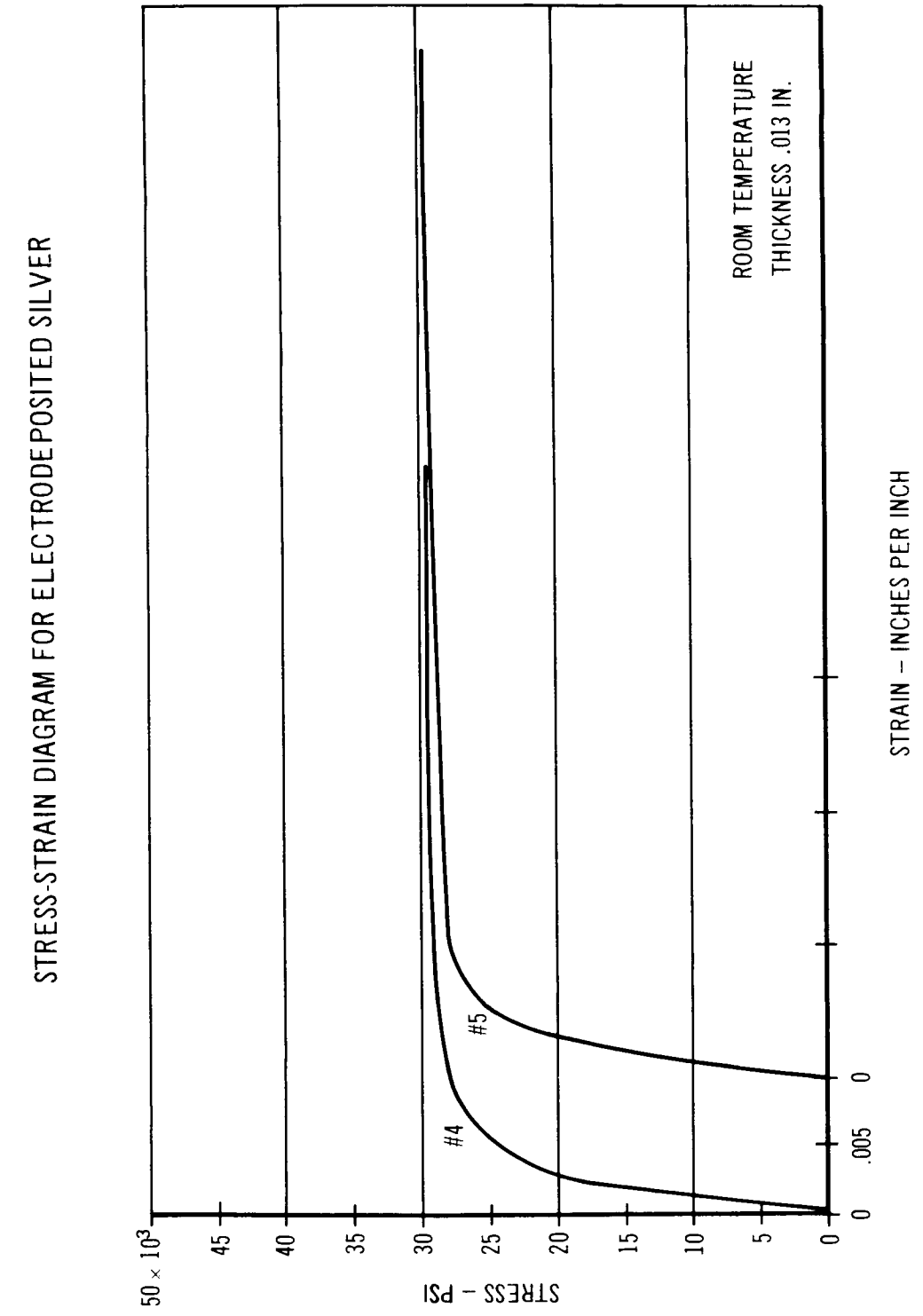

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED SILVER
ROOM TEMPERATURE
THICKNESS .013 IN.
#5
#4
STRESS – PSI
50 × 10³
45
40
35
30
25
20
15
10
5
0
0
.005
0
STRAIN – INCHES PER INCH

FIGURE 2-33

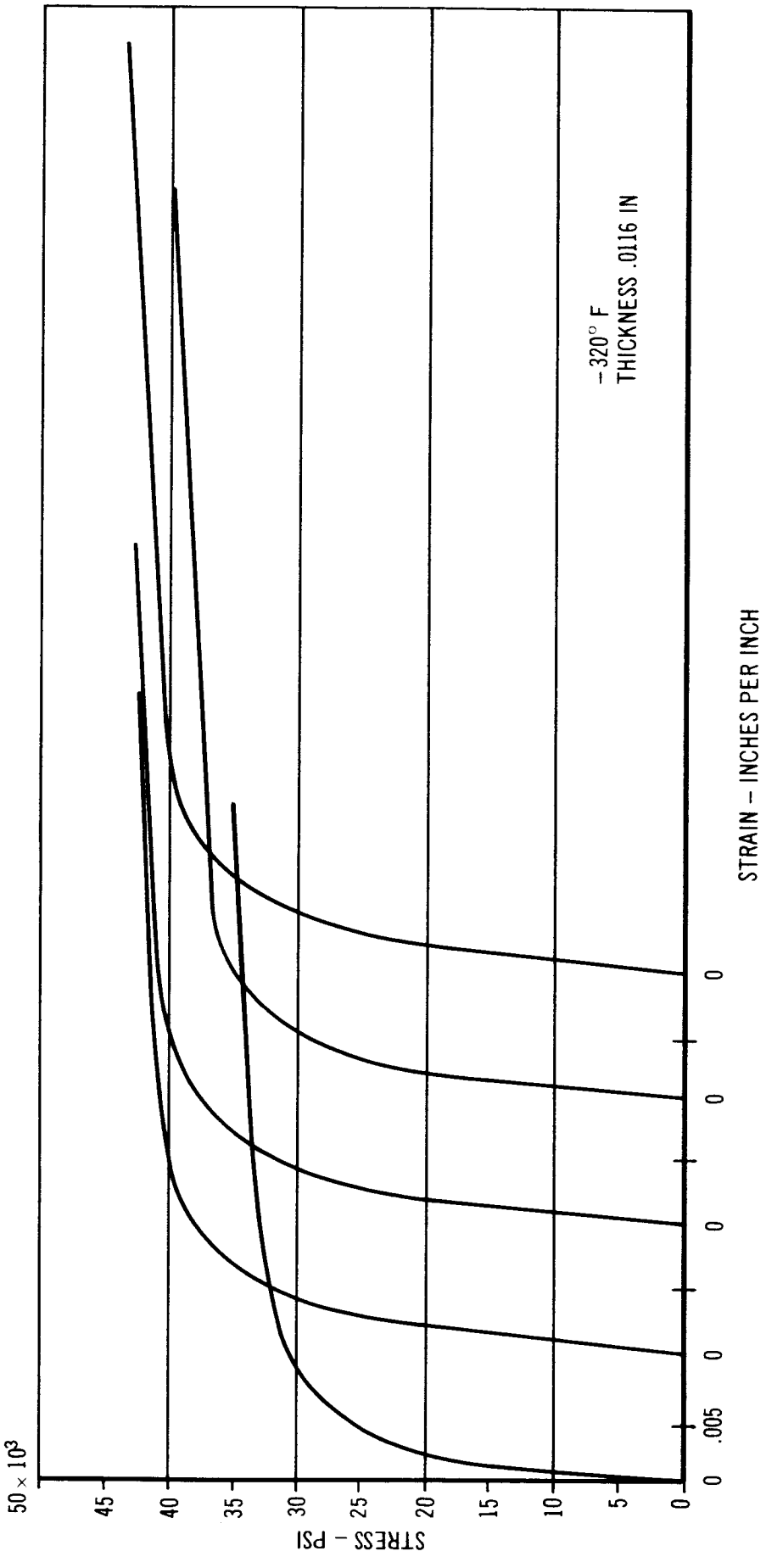

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED SILVER
-320° F
THICKNESS .0116 IN
STRESS – PSI
50 × 10³
45
40
35
30
25
20
15
10
5
0
STRAIN – INCHES PER INCH
0
.005
0
0
0
0

FIGURE 2-34

STRESS-STRAIN DIAGRAM FOR ELECTRODEPOSITED SILVER

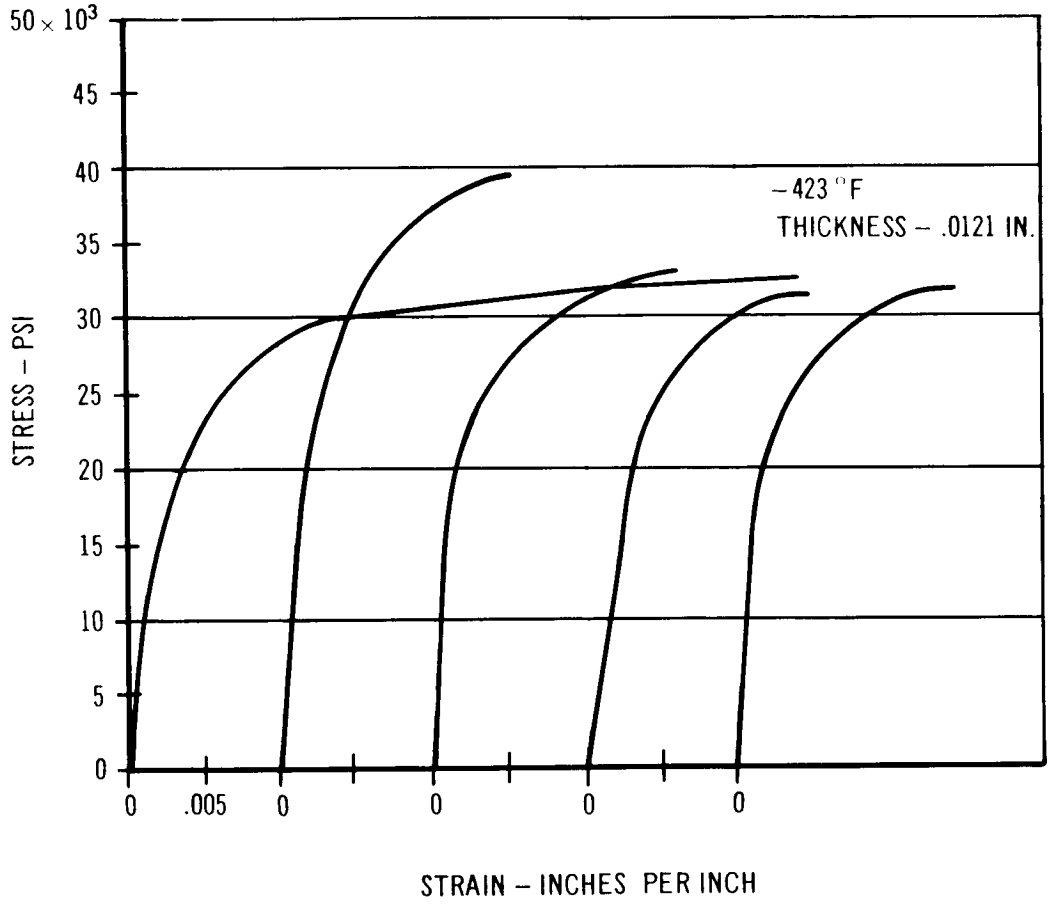


FIGURE 2-35

STRENGTH OF ELECTRODEPOSITED SILVER

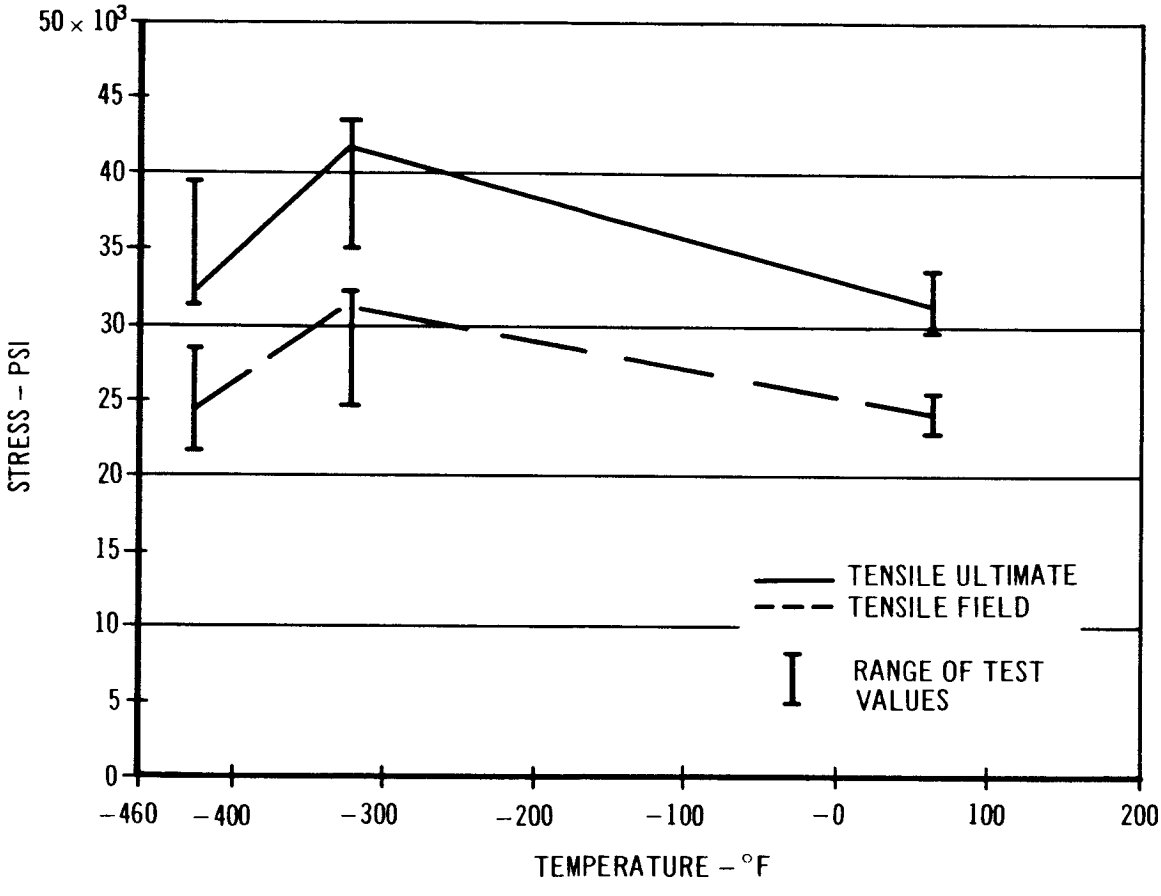


FIGURE 2-36

For the Metallics:

Elongations of all three materials at all temperatures was disappointingly lower than had been expected. None of the values approach those reported in Reference 12 . In fact, the values are less than those of the organic films. The strength of copper as a function of temperature is much lower than that reported in Reference 12; strength of nickel is much higher. It appears that the structure of the electrodeposited metallics and correspondingly the mechanical properties differ considerably from that reported for rolled or wrought material.

2.1.2 Coefficient of Thermal Contraction

Tests were made to determine the coefficient of contraction of the candidate liners between ambient temperature and -423°F. These tests were conducted in the Materials Thermodynamics Laboratory and the Cryogenic Annex.

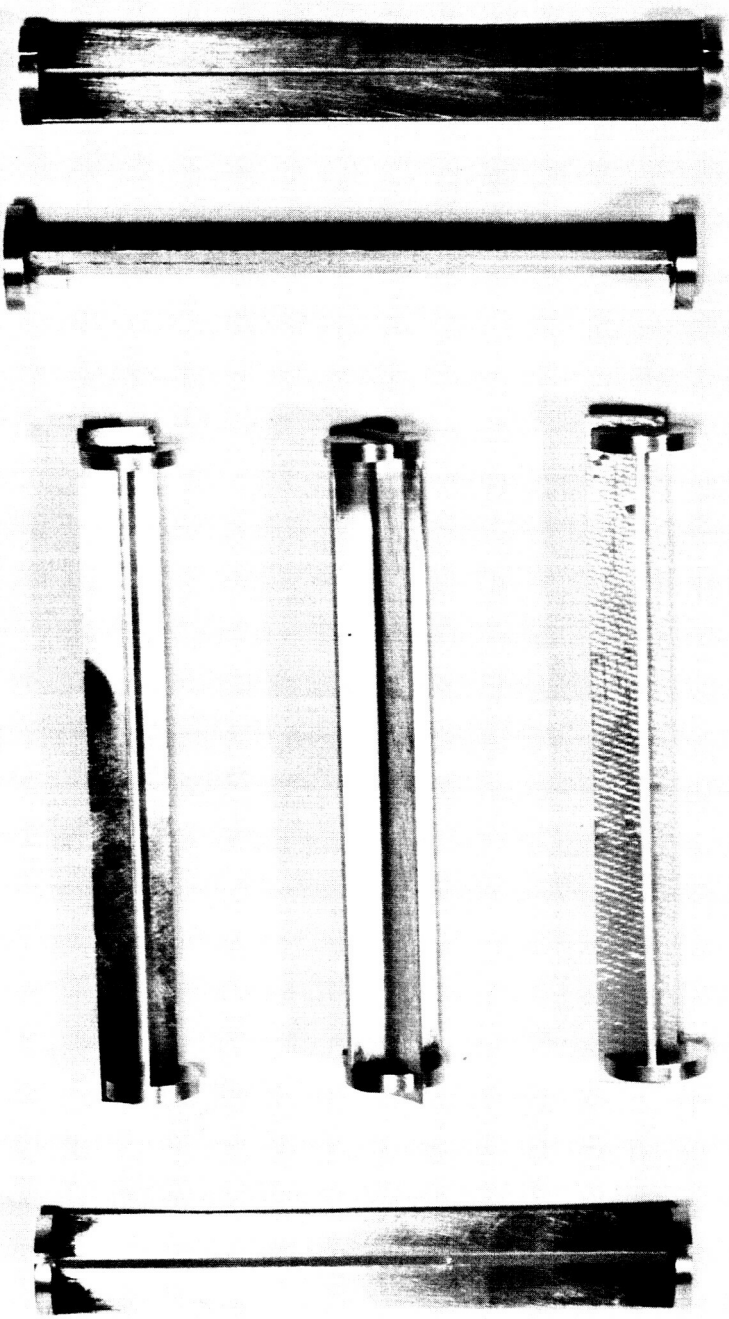
The apparatus and general technique are as described by ASTM D-696-44 Test Method, "Coefficient of Linear Thermal Expansion of Plastics" which utilizes quartz tube dilatometer.

A special holder, Figure 2-37 had been used successfully for thin materials in other programs and it was possible to utilize it for the metallic materials.

Due to the flexibility of the organic materials and the need to support the quartz dilatometer rod, it was necessary to cut the materials into rectangular strips 4-1/2" wide by 6" -24" long (depending upon material), roll then into 1/2" diameter tubes, and tied at two places with Dacron string. The dilatometer and several specimens are shown in Figure 2-38.

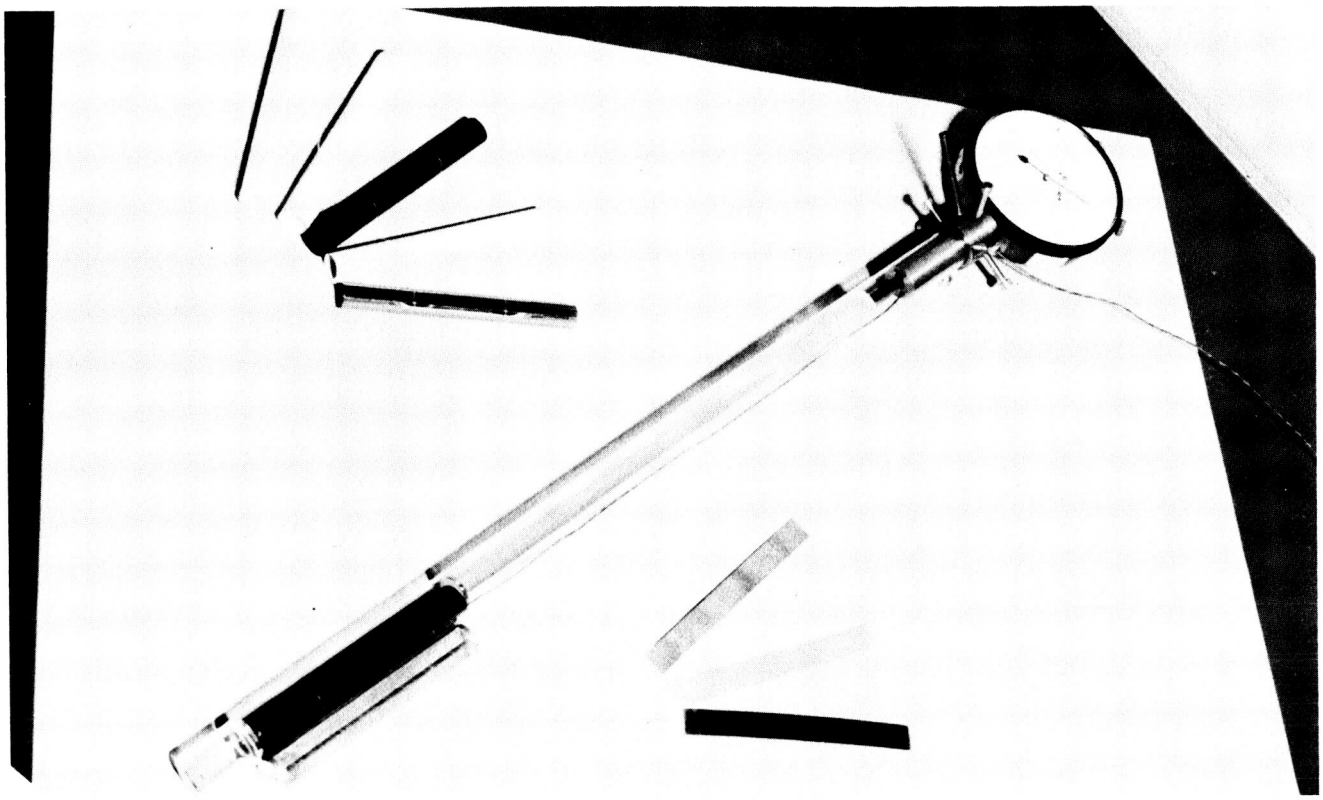
Standard dilatometer procedures were used; upon reaching thermal equilibrium the gage values were noted. Graphical results are shown in Figure 2-39. Tabular results are given in Appendix D.

In order to provide confidence in the results, FEP Teflon was tested by the rolled tube method and the results compared to published data, Reference 29;

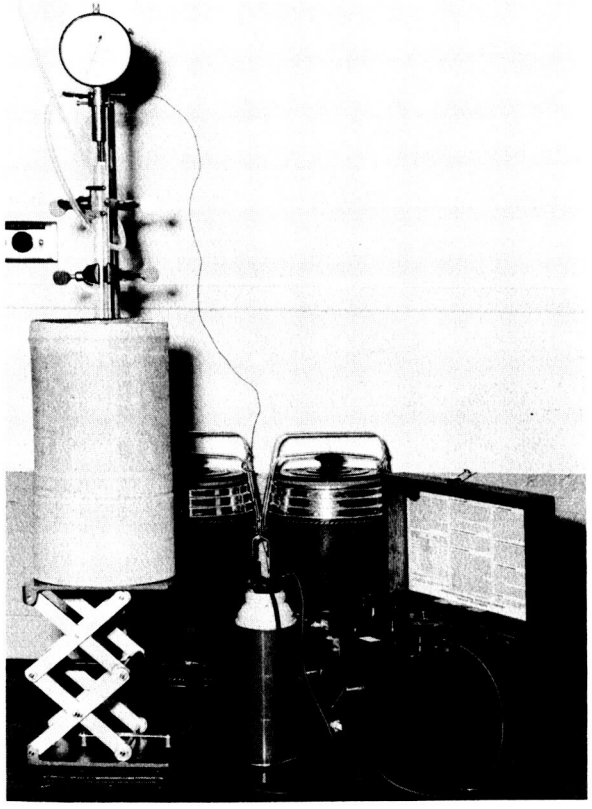


THERMAL CONTRACTION SPECIMENS AND HOLDERS FOR USE WITH QUARTZ TUBE DILATOMETER

FIGURE 2-37



QUARTZ TUBE DILATOMETER



TEST SET-UP

FIGURE 2-38

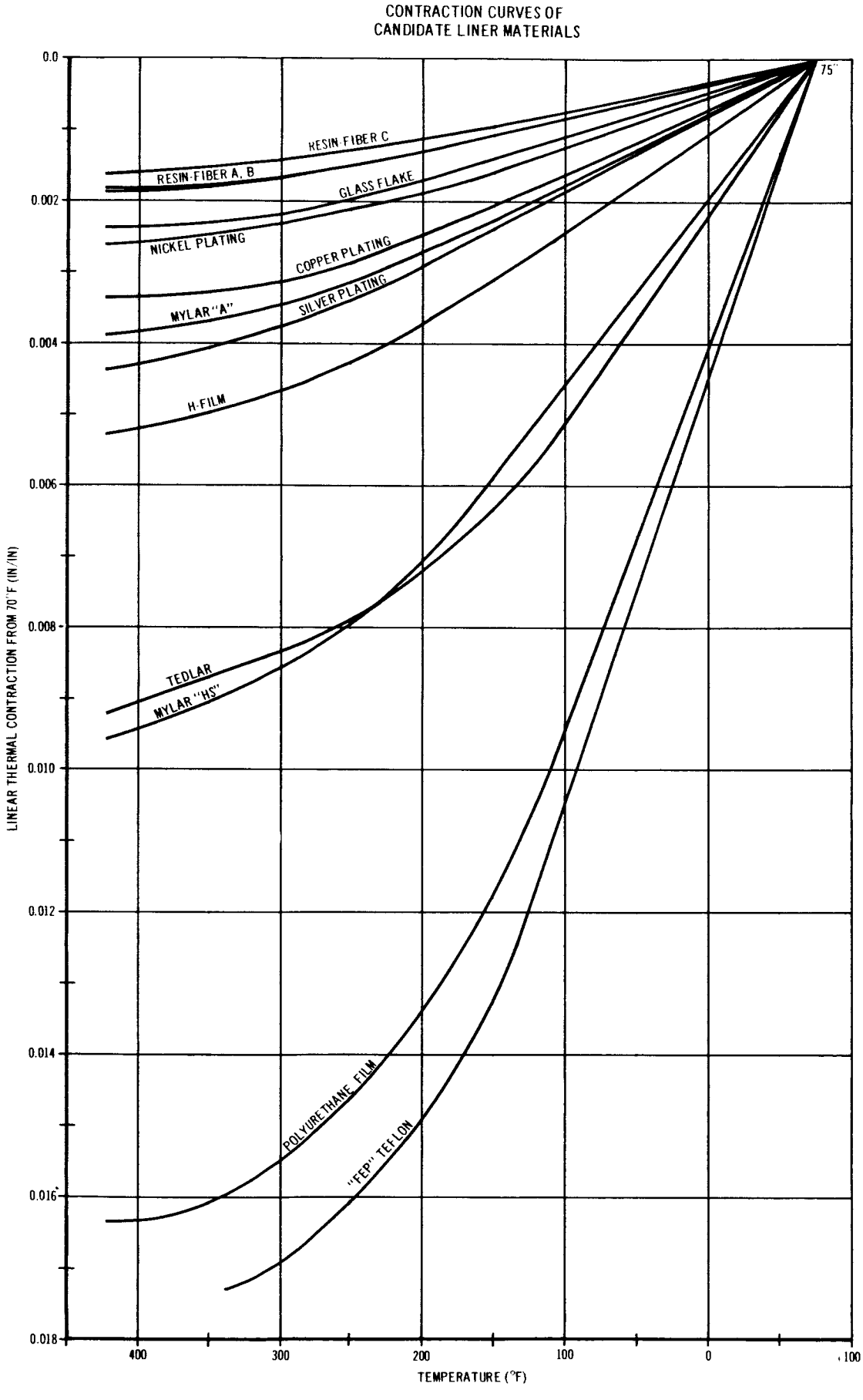

CONTRACTION CURVES OF
CANDIDATE LINER MATERIALS
LINEAR THERMAL CONTRACTION FROM 70°F (IN/IN)
0.0
0.002
0.004
0.006
0.008
0.010
0.012
0.014
0.016
0.018
75°
RESIN-FIBER C
RESIN-FIBER A, B
GLASS FLAKE
NICKEL PLATING
COPPER PLATING
MYLAR "A"
SILVER PLATING
H-FILM
TEDLAR
MYLAR "HS"
POLYURETHANE FILM
"FEP" TEFLON
400
300
200
100
0
-100
TEMPERATURE (°F)

FIGURE 2-39

the values are in agreement. Values for nickel and copper agree with the data of References 3 and 12.

2.1.3 Permeability and Cyclic Tests

Tests were made on the candidate liners to determine their permeability to gaseous nitrogen and hydrogen in the Instrumental Analysis and Nuclear Technology Vacuum Laboratory. Other tests will be made with liquid nitrogen and hydrogen in the Propulsion Laboratory.

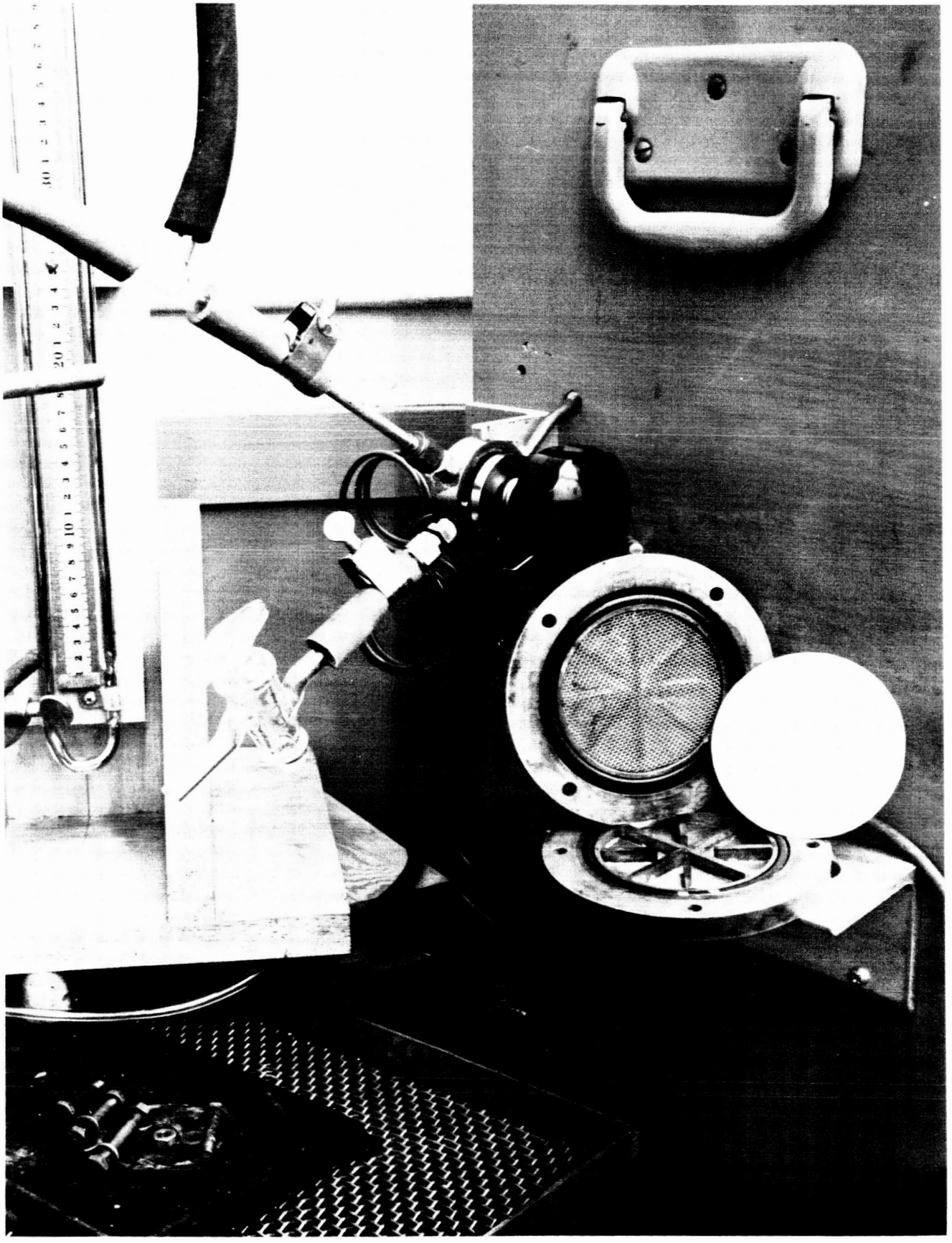
2.1.3.1 Permeability at Ambient Temperature

Unstressed:

In order to provide a relatively simple comparison with other materials that Douglas had evaluated, the candidate liners were tested in an unstressed condition with an ambient temperature permeation cell, **Figures 2-40 and 2-41.** The method of testing was as follows: The specimen to be tested was placed in the cell. The cell consists of two demountable brass discs, each with a cavity 3 inches in diameter and 1/4 inch deep. The specimen was clamped between the two discs and acted as a divider separating the two cavities; "O" ring seals prevented air leakage into the cavities. Air was then evacuated from the collecting cavity and high purity argon was introduced and held at atmosphere pressure. Hydrogen or nitrogen at 10 cubic centimeters per second flowed through the other cavity; thus diffusion occurred from a continuously replenished stream of test gas through the specimen and into the argon rich cavity. Upon reaching a steady-state condition, the mixture in the collecting cavity was flushed out with a stream of argon and introduced into a Linde Molecular Sieve column of a Perkins-Elmer Vapor Fractometer and analyzed. The instrument was standardized by injecting known volumes of gas into the system.

Results of the tests are given in Table **2-2**.

The permeation process is an involved one and subject to many properties of the permeating gas and of the barrier material. Previous work (Reference 7) has shown that the metallics are quite impermeable to the rare gases, while



OPEN DIFFUSION CELL WITH TEST SPECIMEN

FIGURE 2-40

PERMEABILITY MEASURING EQUIPMENT

FIGURE 2-41

TABLE 2-2

UNSTRESSED ROOM TEMPERATURE PERMEABILITY

MATERIAL	HYDROGEN GAS 10^{-9} $cm^3/sec/cc$ at STP	NITROGEN GAS 10^{-9} $cm^3/sec/cc$ at STP
Mylar "HS" (1/2 mil)	37.0 22.0 33.0	3.4 1.7 ---
Mylar "A" (2 mil)	8.6 13.0	1.1 1.6
Polyurethane Seilon UR29E (5 mil)	50.0 54.0	7.1 9.0
Tedlar (2 mil)	5.7 6.9	3.8 2.7
H-Film (1 mil)	23.0 24.0 ----	1.0 1.4 0.9
Silver (10 mil)	Less than 0.04* Less than 0.04*	Less than 0.04** Less than 0.04**
Copper (5 mil)	Less than 0.02* Less than 0.02*	Less than 0.02** Less than 0.02**
Nickel (7 mil)	Less than 0.03* Less than 0.03*	Less than 0.03** Less than 0.03**

* No detectable Hydrogen for given test time. Values are for limit of apparatus for given time period.

** Values for Nitrogen must be lower than those for Hydrogen.

Glass flake material will be tested at a later date.

polymers will always show evidence of permeation. The results of the present tests substantiate those views. Theoretically, the permeation rate should be a function of molecular weight in accordance with Grahams Law, i.e.

$$\frac{\text{Permeation of Hydrogen}}{\text{Permeation of Nitrogen}} = \sqrt{\frac{\text{Molecular Weight } N_2}{\text{Molecular Weight } H_2}}$$

$$= \frac{3.7}{1}$$

However, this does not hold true for the results tabulated here nor for most of the information in the literature, References 8,9,18,23,32-34 . Other theoretical work suggests that molecular diameter and velocity are the governing factors; this is also not corroborated by experimental data (various properties of pertinent gases are given in Appendix E). It appears that the permeation process is so complex that it is almost impossible to predict quantitative values from one gas to another; qualitatively, the results given here agree with that of the manufacturer's literature and recent work of Bailey (Reference 35). Unpublished work of Bailey also suggests that the complete permeation process takes place in amorphous regions of the polymeric films. This would tend to explain the higher permeability for Mylar "HS" vs. "A" material, although pinholes probably existed in the very thin (1/2 mil) "HS" material.

Biaxially Stressed:

In order to evaluate ambient temperature permeability on stressed samples, the Douglas biaxial test specimen (Figure 2-42) is being used. This test has been designed to provide a correlation between unstressed and stressed conditions.

Fabrication of each specimen is as follows and is illustrated in Figures 2-43, to 2-46: The cylinders are fabricated on segmented aluminum mandrels. The mandrel is thoroughly cleaned and a mold releasing Teflon spray is applied. The mandrel is lightly buffed after each application if the

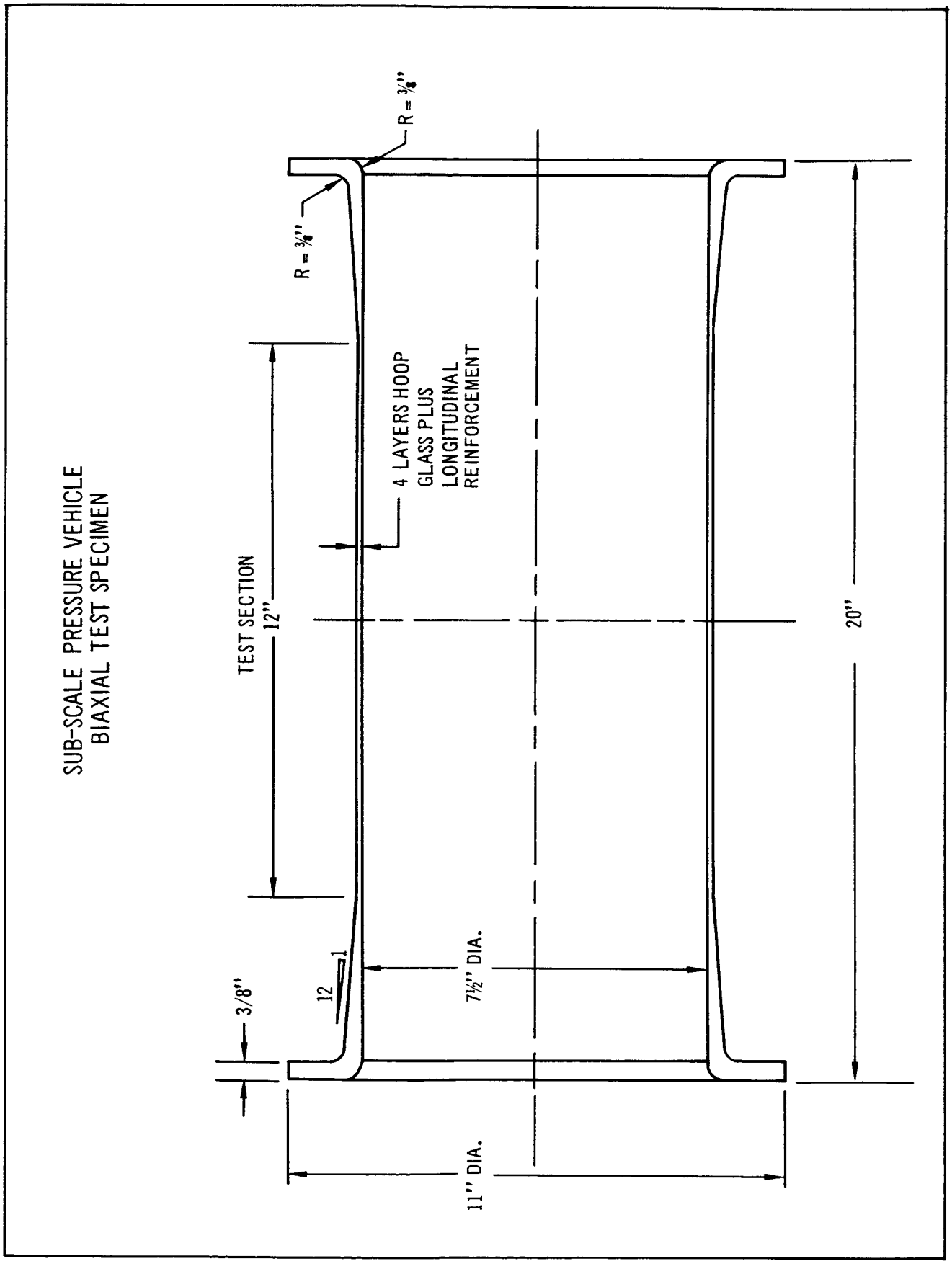
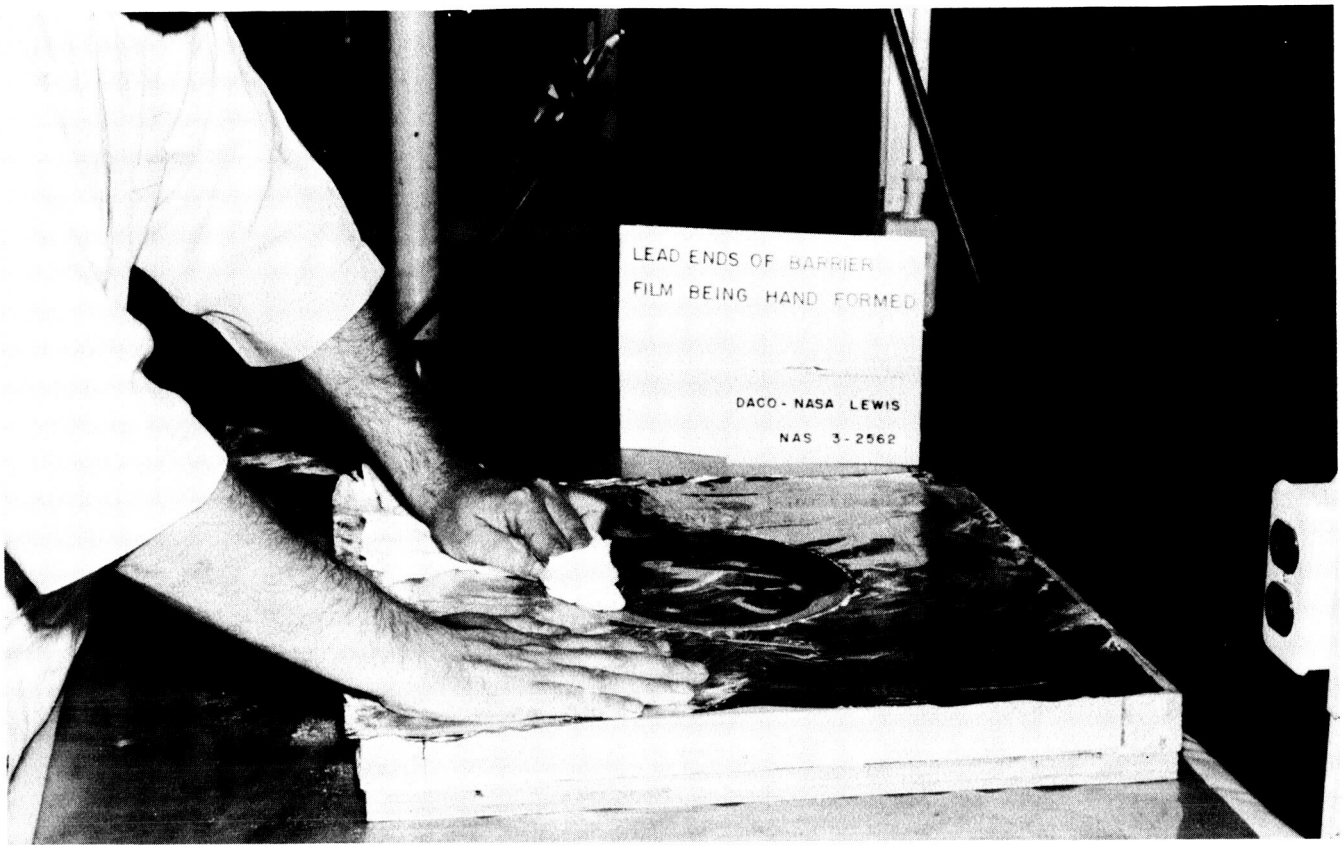
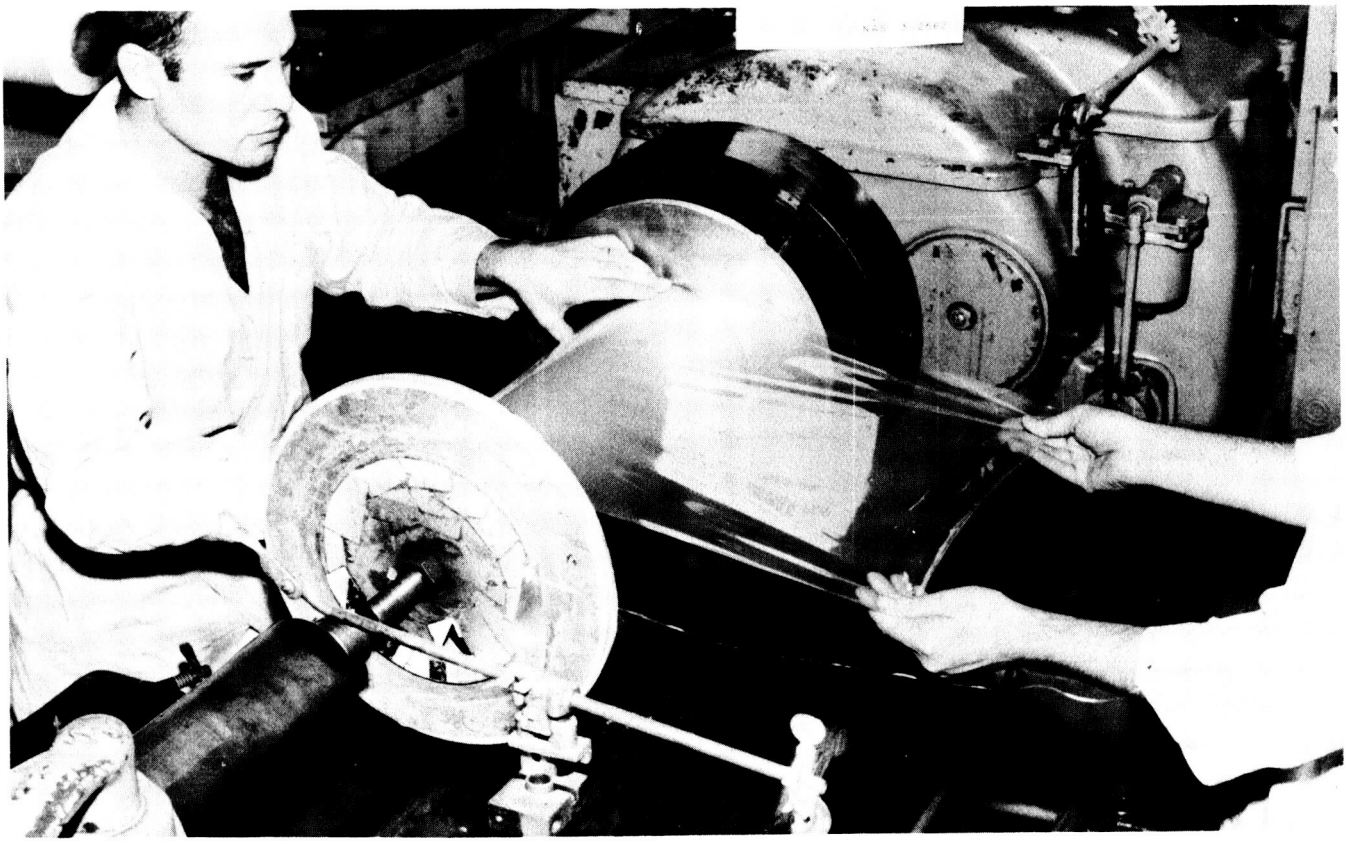

SUB-SCALE PRESSURE VEHICLE
BIAXIAL TEST SPECIMEN
R = 3/8"
R = 3/8"
4 LAYERS HOOP
GLASS PLUS
LONGITUDINAL
REINFORCEMENT
TEST SECTION
12"
20"
3/8"
12
1
7½" DIA.
11" DIA.

FIGURE 2-42

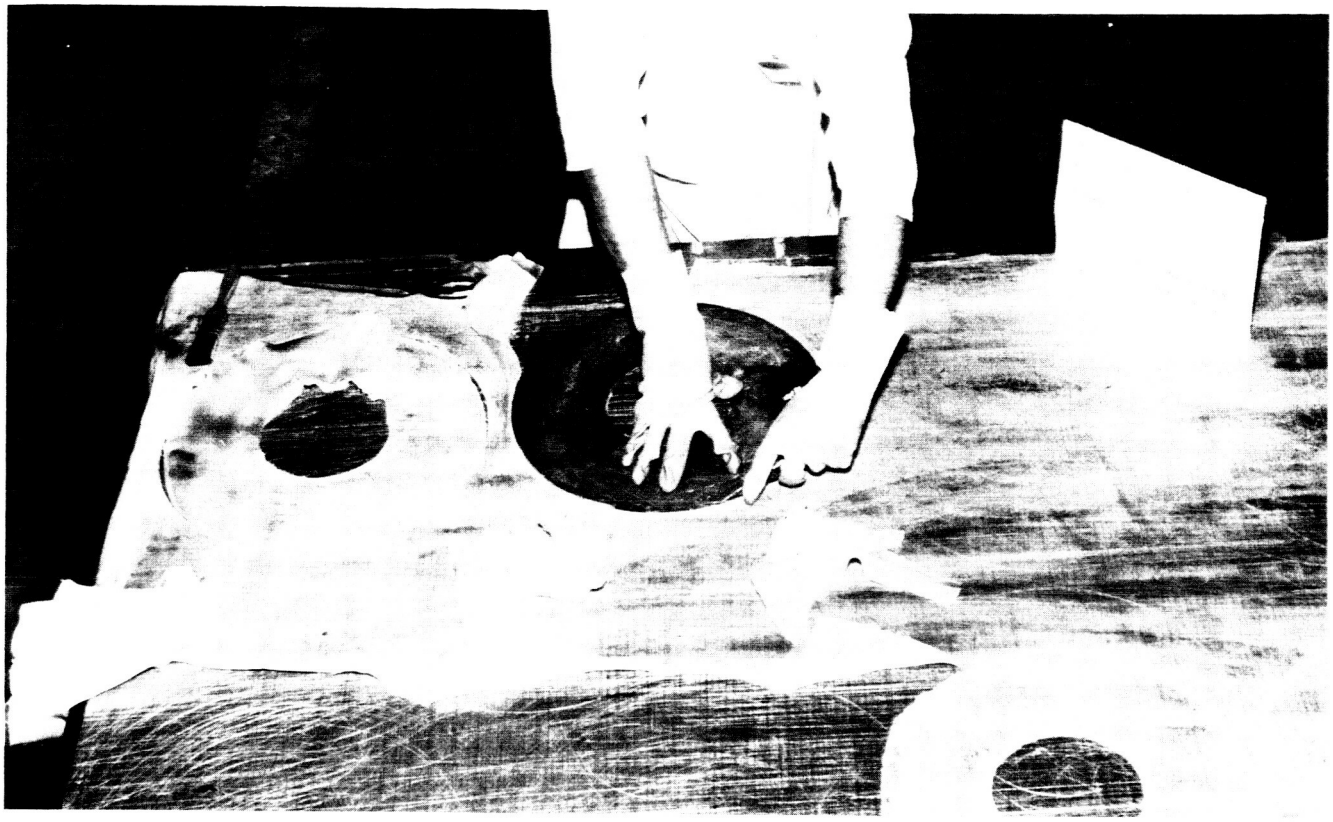


1. FORMING LEAD END SEALS.

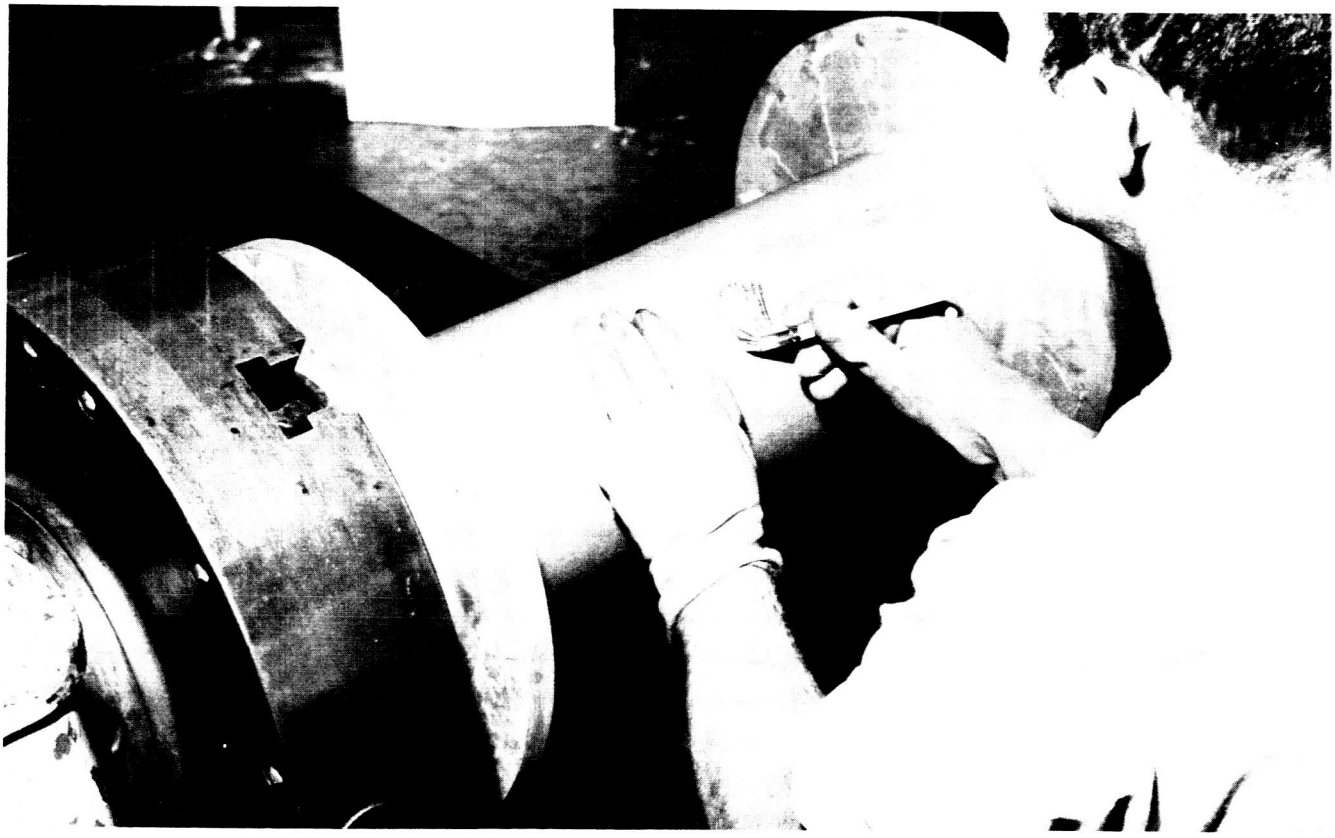


2. WRAPPING MYLAR "A" BARRIER FILM

FIGURE 2-43

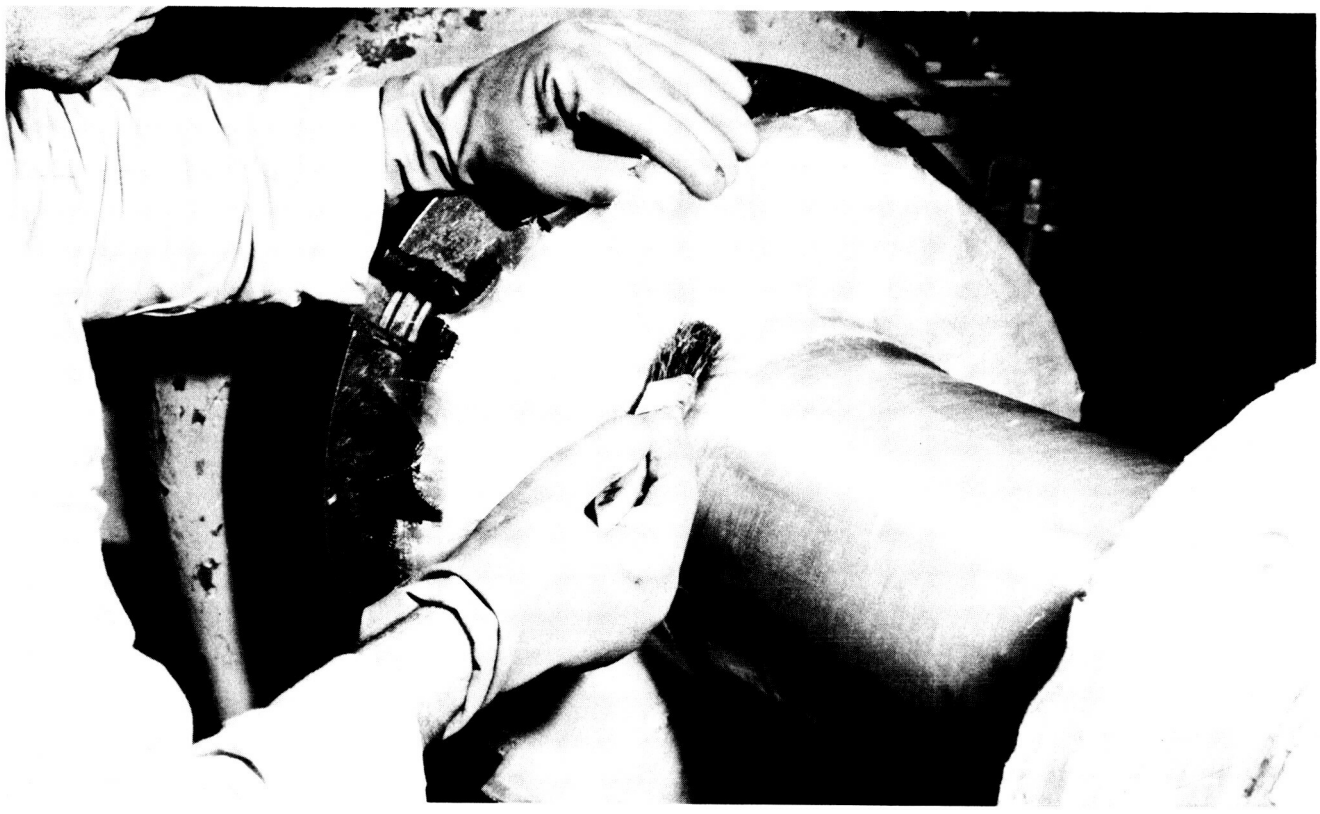


3. IMPREGNATING AND CUTTING OF FIBERGLASS CLOTH

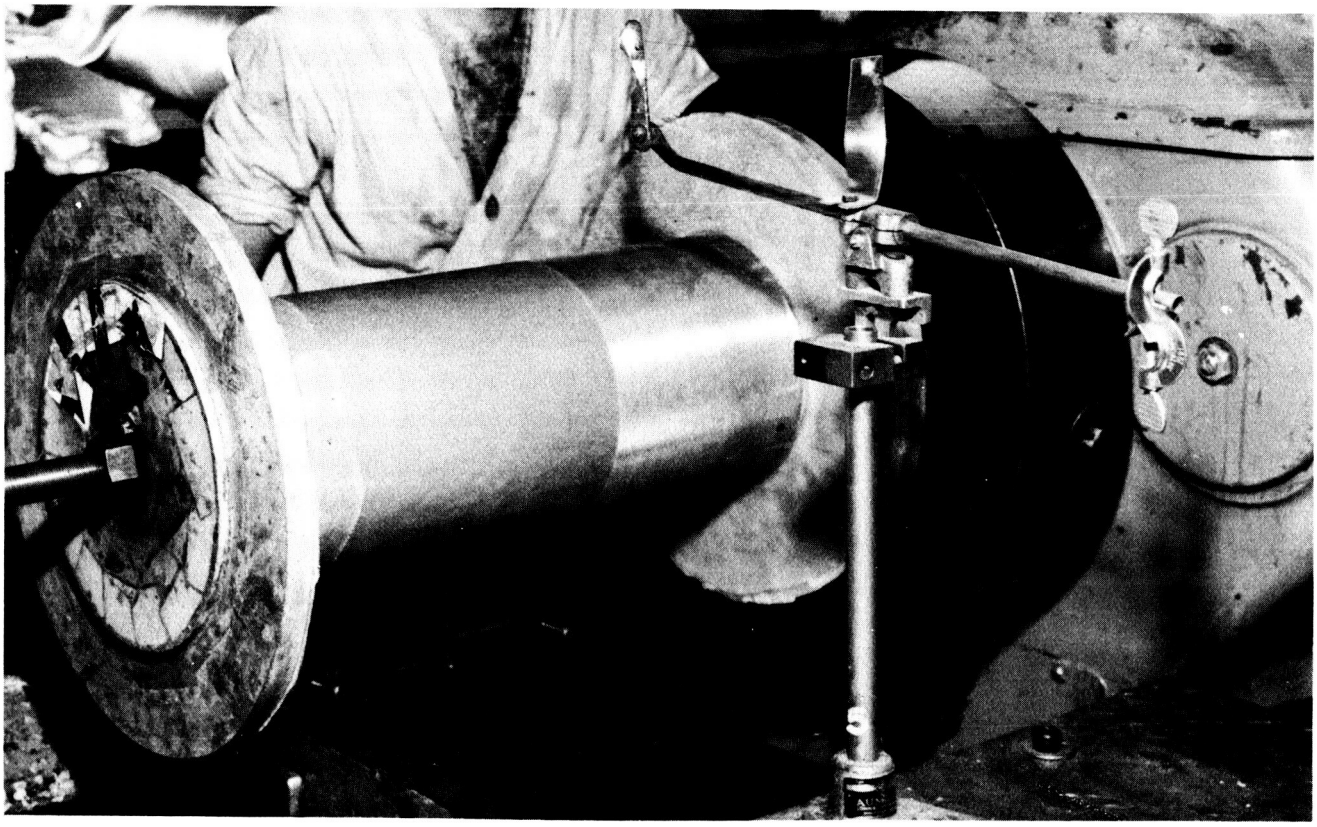


4. LAY-UP OF STYLE 143 FIBERGLASS CLOTH

FIGURE 2-44

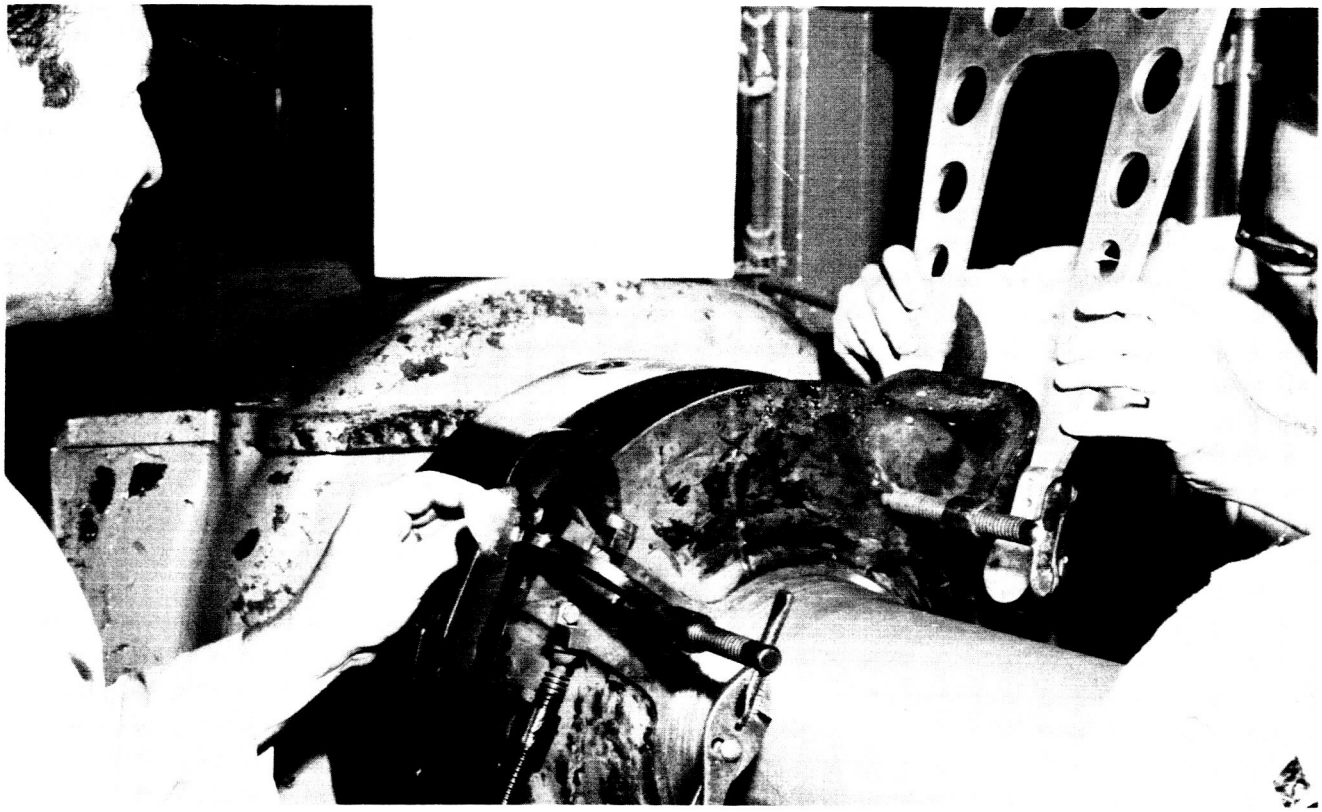


5. LAY-UP OF STYLE 181 CLOTH AT ENDS OF CYLINDER.



6. CIRCUMFERENTIAL WINDING – FIRST WRAP

FIGURE 2-45



7. POSITIONING OF CLAMP RINGS

8. REMOVING MANDREL END PLATE

FIGURE 2-46

spray, generally three to five coats are considered sufficient.

Next the liner is formed. If the liner is to be a metallic foil, the mandrel is shipped to the vendor where he applies the approved thickness of electrodeposited metal. The liners are certified to be pit or void free. The liner extends beyond the cut-off line of the part, and is then trimmed back to this point. A surface treatment of the liner is made with a mixture of aluminum oxide and distilled water to prepare the metallic surface for bonding to the fiberglass. Adhesive (described below) is applied to the liner just prior to fabricating the **fiber glass shell.**

If the liner is to be an organic film, a different procedure is followed. The gasketing and seal of the cylinder ends is made of 8-mil lead. The lead must be formed from lead foil sheeting using a female plaster mold. The plastic film is cut in a length necessary to wrap the cylindrical section with three layers of film. The film must be as wide as the length of the cylindrical section. Adhesive is applied to the surface of the film on one side covering all of the length except an area long enough to wrap the last wrap around the cylinder. The liner is wrapped around the cylindrical section of the mandrel with the uncoated side for the top layer. It is then lag-wrapped with removable shrink tape to allow the adhesive to cure under pressure. After the adhesive has cured and the shrink tape removed, adhesive is applied to the liner surface just prior to wrapping with **fiber glass.**

The fabrication procedure involves trimming the fiberglass cloth to size and preimpregnating the cloth with the desired resin system. A wet lay-up of the cloth is accomplished while interspersing circumferential wraps of single end S994/HTS S &Z twist **fiber glass;** then clamp rings are installed to apply pressure to the flange area. The part then goes through its proper cure cycle. Finally the segmented mandrel is removed and the part is trimmed, bagged in polyethelyene bags, and marked. The part is now ready for set-up and instrumentation for test.

Adhesive tests were made for three of the films (Mylar, Tedlar, and H-Film) to be used in the program. From available information (References 2, 36-40), adhesives chosen were Narmco 7343/7139, polyurethane based adhesive, and Minnesota Mining and Manufacturing EC2216 B/A, a modified epoxy adhesive. The plastic films were bonded to stainless steel sheet shear pads and then bonded together with the selected adhesive, Figure 2-47 Results were as follows (values are averages of 3 specimens at -423°F and 2 specimens at room temperature) (Reference 41):

TENSILE-LAP SHEAR IN PSI

FILM	THICKNESS	ADHESIVE	RT	-423°F
Mylar	2 mil	Narmco 7343/7139	620 psi	903 psi
Mylar	2 mil	EC 2216 B/A	695	626
Tedlar	1 mil	Narmco 7343/7139	880	1946
Tedlar	1 mil	EC 2216 B/A	1245	772
H-Film	2.5 mil	Narmco 7343/7139	555	2089
H-Film	2.5 mil	EC 2216 B/A	1320	2663

Due to the apparent applicability of the elevated cure resin system, Epi Rez 5101/Apco 322, as indicated from the uniaxial cyclic testing (Section 2.2.2) adhesive tests were also made to determine the effect of the elevated cure cycle upon the selected adhesives. The cure cycle was: ambient temperature gel for 8 hours, 150°F for 1 hour, and 250°F for 3-1/2 hours. Results were as follows:

TENSILE-LAP SHEAR IN PSI

FILM	THICKNESS	ADHESIVE	RT	-423°F
Mylar	2 mil	Narmco 7343/7139	705 psi	843 psi
Mylar	2 mil	EC 2216 B/A	1048	911
Tedlar	1 mil	Narmco 7343/7139	705	2866
Tedlar	1 mil	EC 2216 B/A	1275	1410
H-Film	2.5 mil	Narmco 7343/7139	729	1076
H-Film	2.5 mil	EC 2216 B/A	1424	1330

ADHESIVE LAP-SHEAR TENSILE TEST SPECIMEN

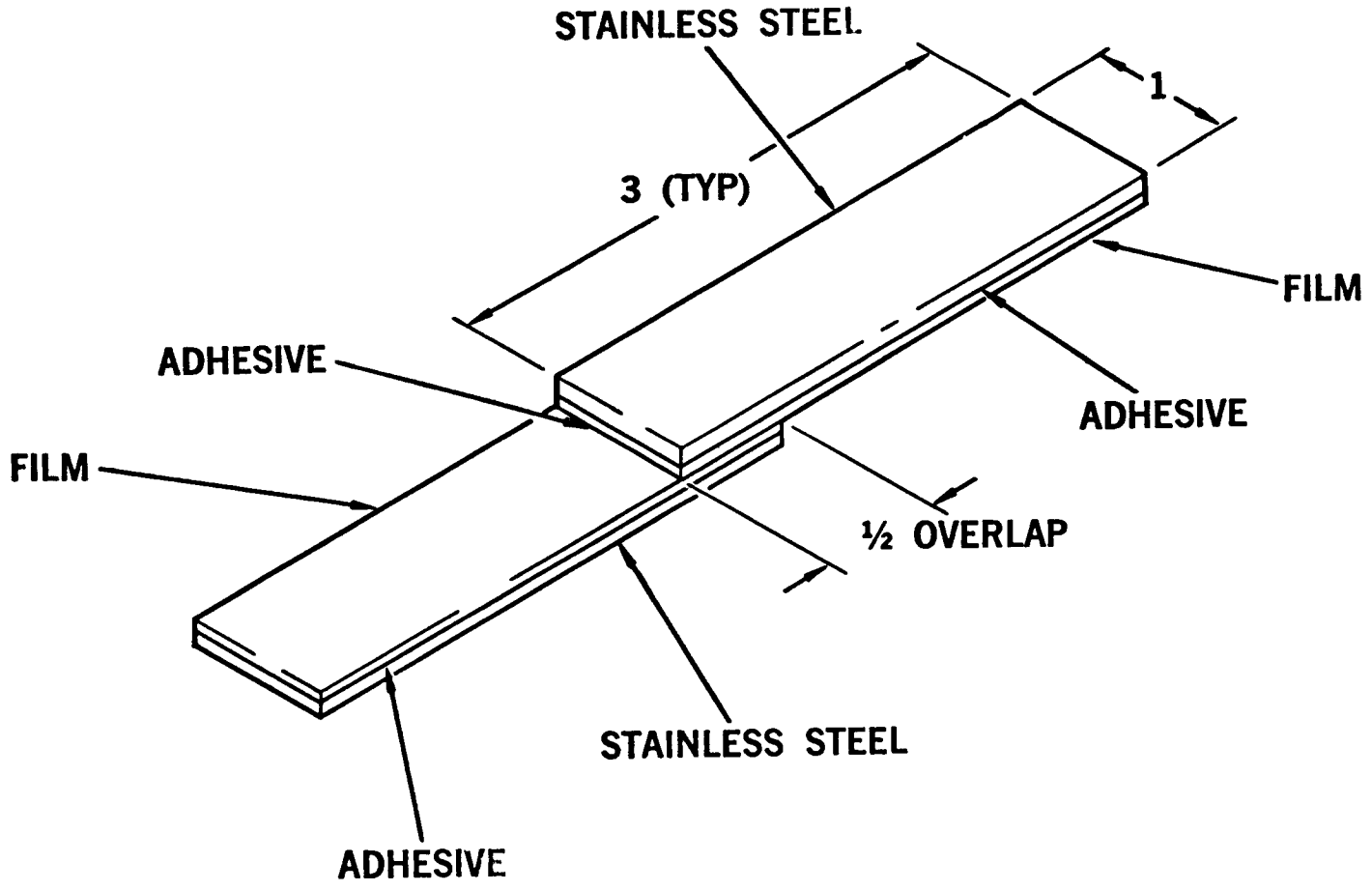


FIGURE 2-47

All of the test specimens have been made for this part of the investigation. The first part of this evaluation requires a burst test to determine the ultimate strength of the structural wall; then the remaining specimens are pressurized to 60% of the ultimate load, permeability data taken, and subsequently cyclic pressurized to failure. Two attempts to burst a nickel-lined specimen failed due to an inadequate seal of the ends and apparently a work-hardening rupture of the liner. A premature burst of a Seilon UR29E polyurethane line specimen negated any information other than the fact that failure occurred in the hoop windings, as desired; specimen was not instrumented at time of burst. A second Seilon UR29E polyurethane lined specimen was instrumented and successfully tested. A schematic of the test set-up used for the burst test is shown in Figure 2-48 . The permeability tests will use a test set-up as shown schematically in Figure 2-49 .

Difficulty has been experienced in checking out and utilizing the newly fabricated vacuum chamber and vacuum system. The Veeco Residual Gas Analyzer, which will be used to make the permeability measurements, must have a pressure less than 10^{-4} Torr at the sampling point. Since it was not possible to obtain pressures less than 10^{-1} Torr in the chamber for a straight-through system, a throttling valve has been installed and it appears that the system will function satisfactorily.

2.1.3.2 Permeability at -320°F and -423°F

Since this will be the most significant testing during Phase I, it will also employ the high performance technique of biaxial testing to give reliable, accurate information for proper evaluation of liners.

The testing with the biaxial test specimen provides for complete evaluation of a candidate liner material under stress at the desired cryogenic temperature, subject to representative internal pressures using liquid nitrogen or liquid hydrogen as the pressurizing medium.

Test will be made with the biaxial test specimen, the test chamber previously described, and a test set-up shown schematically in Figure 2-50.

PRESSURE VESSEL TEST SCHEMATIC FOR AMBIENT TEMPERATURE BURST TESTS

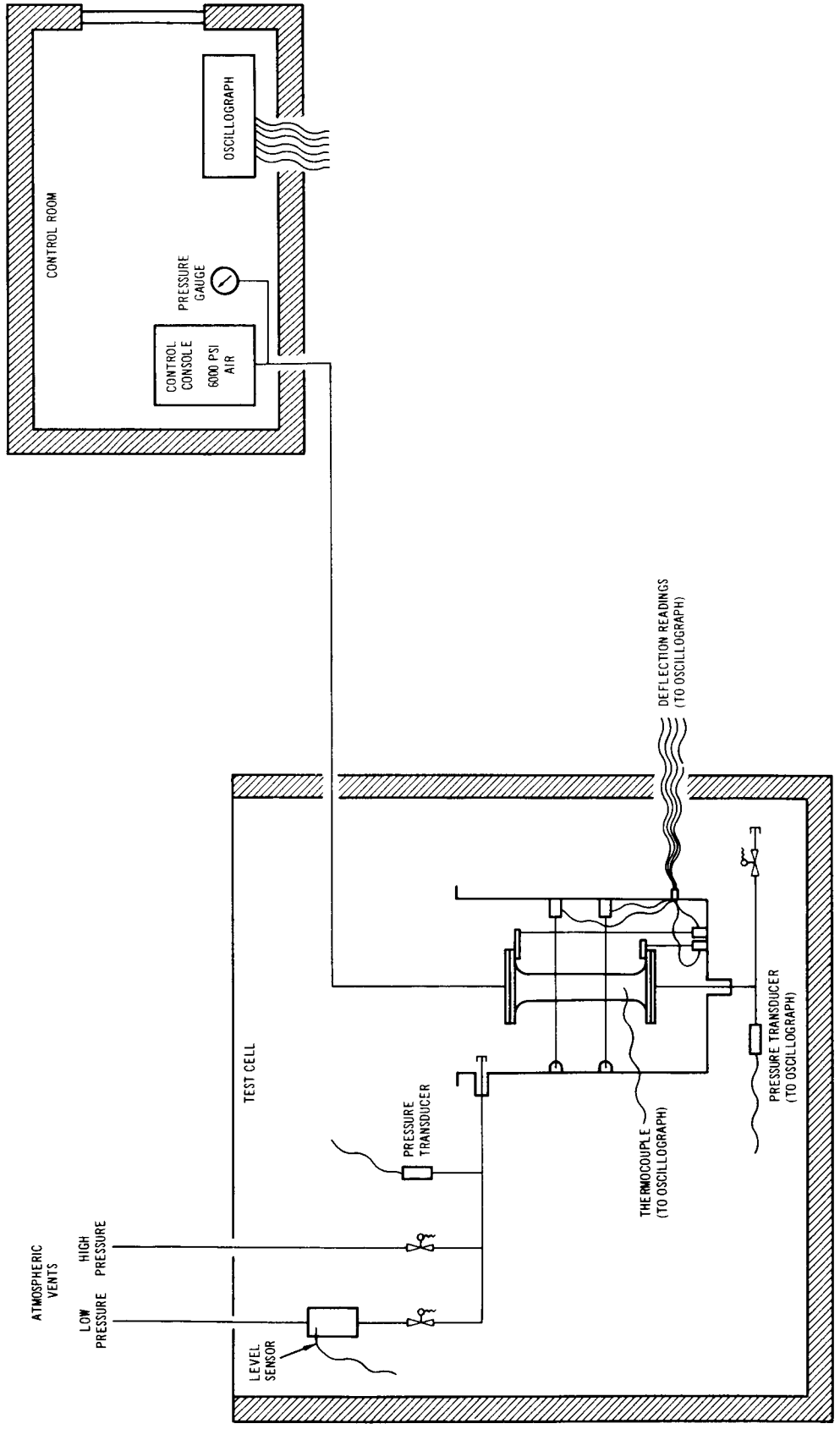


FIGURE 2-48

PRESSURE VESSEL TEST SCHEMATIC FOR AMBIENT TEMPERATURE PERMEABILITY AND CYCLIC TESTS

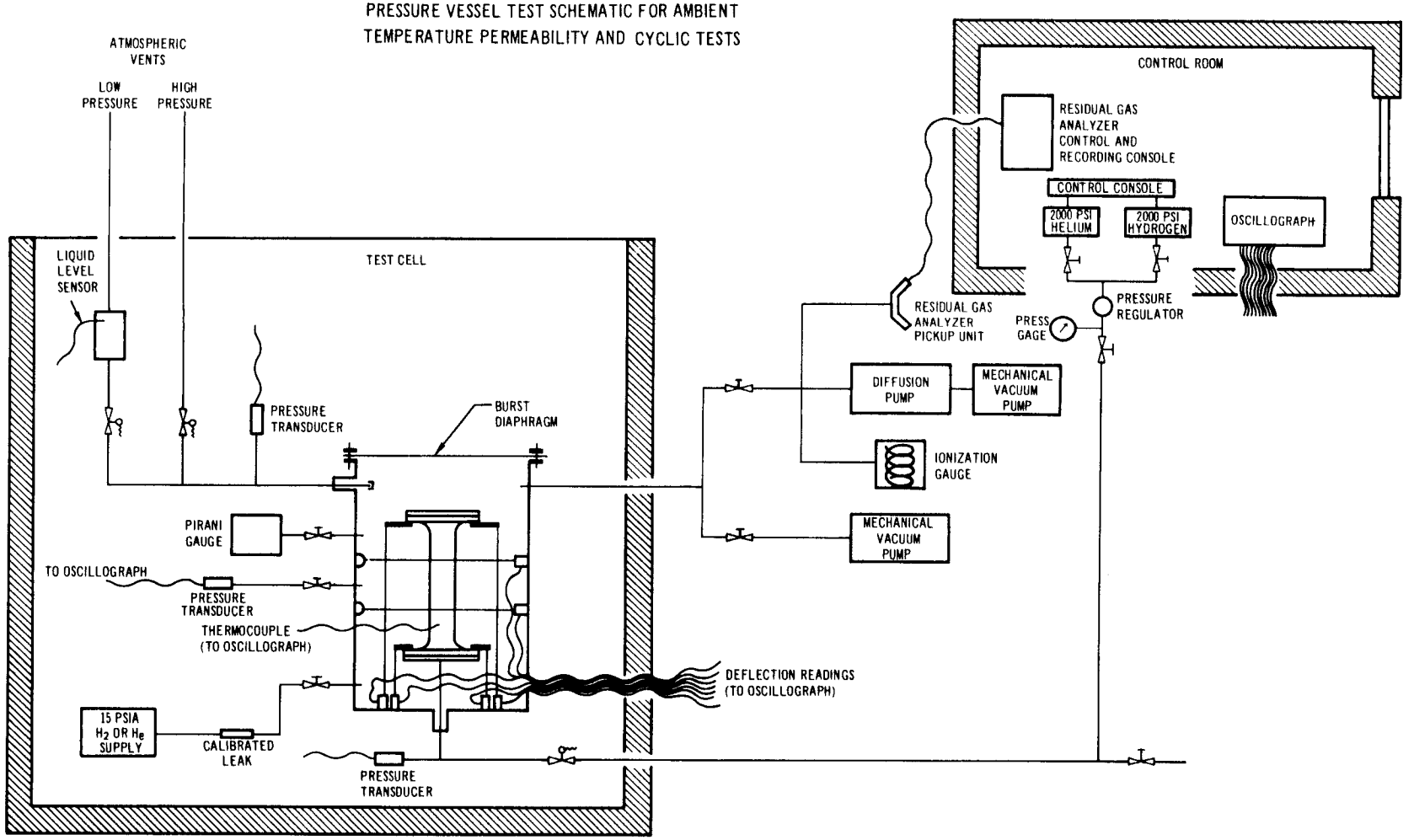


FIGURE 2-49

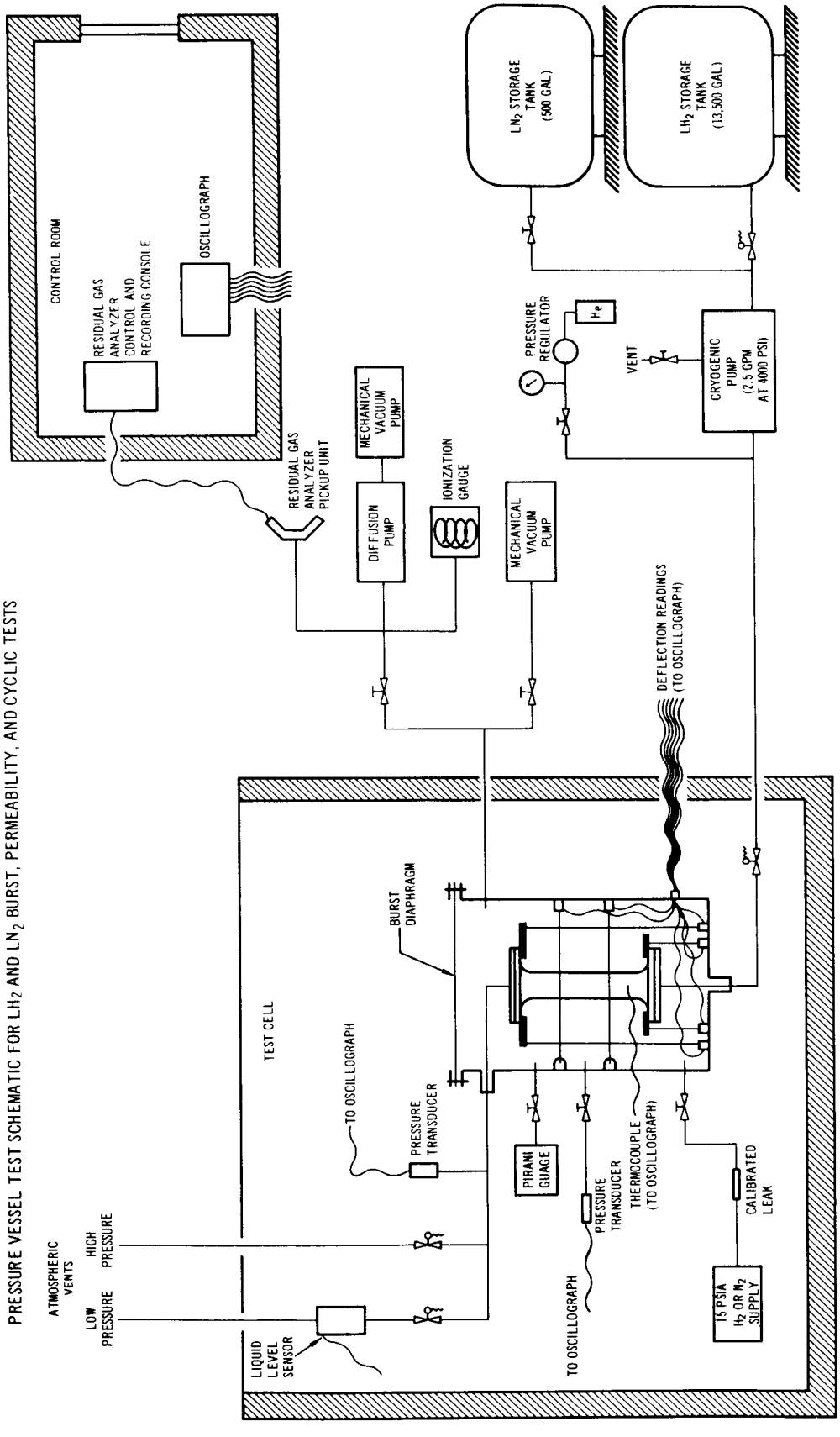

PRESSURE VESSEL TEST SCHEMATIC FOR LH_2 AND LN_2 BURST, PERMEABILITY, AND CYCLIC TESTS
CONTROL ROOM
RESIDUAL GAS ANALYZER CONTROL AND RECORDING CONSOLE
OSCILLOGRAPH
RESIDUAL GAS ANALYZER PICKUP UNIT
MECHANICAL VACUUM PUMP
DIFFUSION PUMP
IONIZATION GAUGE
MECHANICAL VACUUM PUMP
LN_2 STORAGE TANK (500 GAL)
LH_2 STORAGE TANK (13,500 GAL)
PRESSURE REGULATOR
He
VENT
CRYOGENIC PUMP (2.5 GPM AT 4000 PSI)
DEFLECTION READINGS (TO OSCILLOGRAPH)
TEST CELL
BURST DIAPHRAGM
TO OSCILLOGRAPH
PRESSURE TRANSDUCER
PIRANI GUAGE
PRESSURE TRANSDUCER
THERMOCOUPLE (TO OSCILLOGRAPH)
CALIBRATED LEAK
ATMOSPHERIC VENTS
LOW PRESSURE
HIGH PRESSURE
LIQUID LEVEL SENSOR
TO OSCILLOGRAPH
15 PSIA H_2 OR N_2 SUPPLY

FIGURE 2-50

The permeability test pressure will be 60% of the ultimate, based on results from the fiber glass/composite properties tests. This pressure has been chosen because it is representative of testing which will be performed later in the program.

Since a standard of permeability has not been set, measurements of the diffused gases will be made either with a residual gas analyzer or a gas chromatograph, whichever meets the actual test requirements. It is felt that no problem will be met in holding a vacuum in the vacuum chamber due to the cryopumping of the test specimen.

A newly acquired cryogenic pump is being installed for use in this program.

2.1.3.3 Cyclic Testing

The initial permeability tests will be the first cycle of this part of the program. The permeability specimens will be cycled at a rate to be determined by mutual consent with the NASA-Lewis Program Director. The specimens will be fully instrumented and cycled at 60% of the ultimate load until failure occurs. Cycling tests will be made at room temperature, -320°, and -423°F. Permeability rates during cycling will be recorded, if possible.

2.2 FIBER GLASS/RESIN COMPOSITE INVESTIGATION

The tensile strength, yield strength, modulus of elasticity, ultimate elongation, and coefficient of thermal contraction are being evaluated for each candidate system.

2.2.1 Candidate Materials

Owens-Corning S994-fiber glass is being evaluated with two room-temperature curing resin systems and one elevated-temperature curing resin system. The S994-glass is being used since it represents the most advanced product available and its mechanical properties are superior to E/HTS glass. (Reference 42).

Douglas has evaluated many resin systems for structural plastics applications.

The requirements differ for each resin system application. For instance, filament-wound structures which undergo cycling grow considerably in diameter. When loaded, the hoop load is usually large and if allowed to remain, permanent deformations are produced due to plastic flow (Reference 43). For such a structure, the elastic limit of the resin at the use temperature should not be exceeded by the anticipated loads, and good hysteresis characteristics must be present in the resin. A highly cross-linked system with no plasticizing diluents is essential.

The epoxy resins were selected by chemical constitution so that they contained only a polyfunctional monomer. Deleterious constituents in epoxy resins to be avoided are monofunctional epoxy compounds such as phenyl glycidyl ether which, if present in quantities above 1%, will drastically reduce the physical properties, particularly the impact strength of the cured system. In addition, hardener choice was based on aromatic amines, straight or eutectic blends, which would impart flexibility to the cured system and react totally. This is important because only if total or near total reactions are accomplished, will the systems be flexible at cryogenic temperatures. (References 44-46).

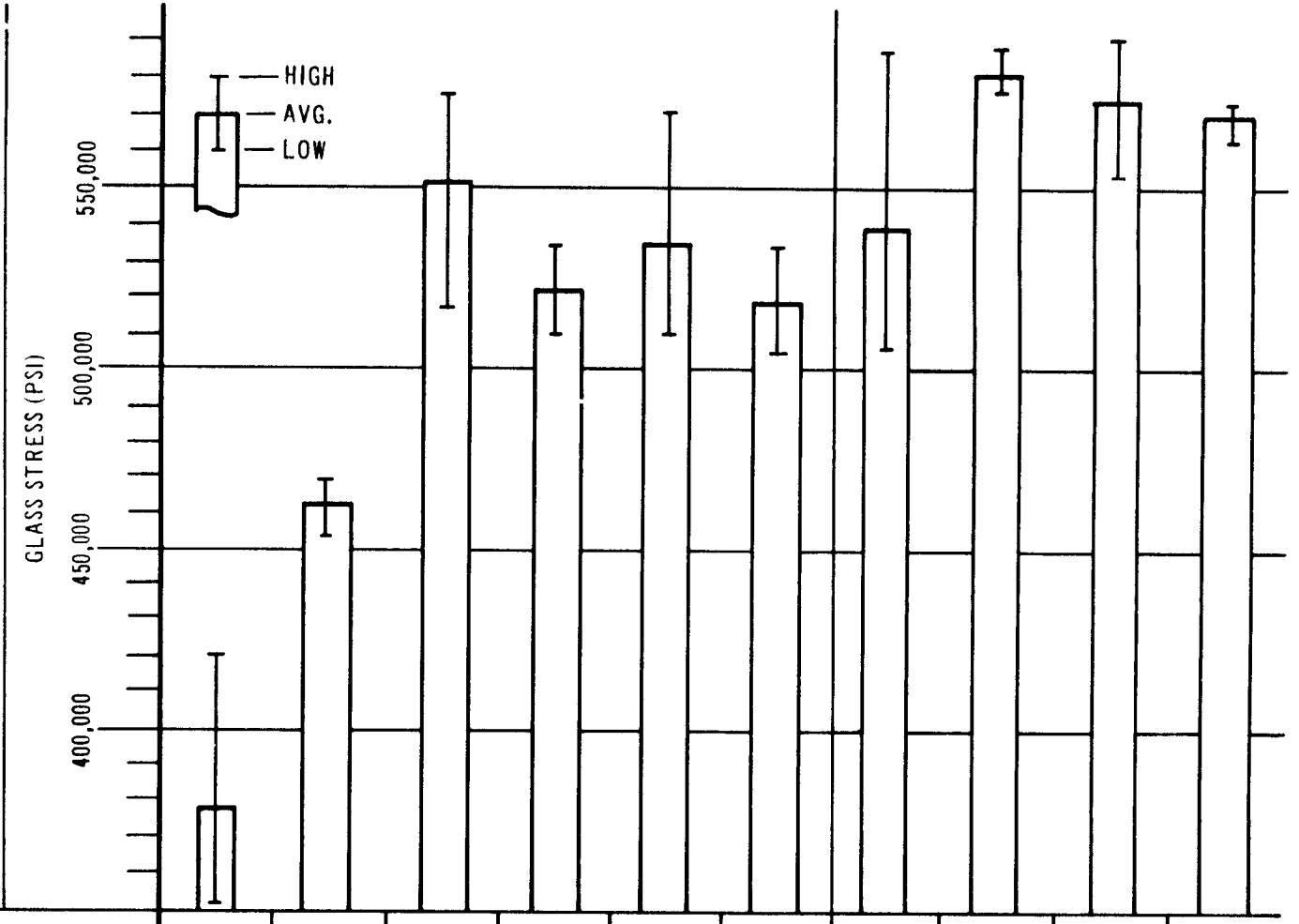
Ten resin systems with the characteristics outlined above were selected for evaluation at cryogenic temperatures. The evaluation was made with 6-inch diameter NOL rings. The resin systems were tested in liquid nitrogen with the split disc apparatus. The test resins and results are shown in Figure 2-51. On the basis of this evaluation, three resin systems were chosen for the program:

A.	ERLA-0510/22L 0803	(Room Temperature Cure)
B.	EPI-REZ 510-5042/EPICURE 841	(Room Temperature Cure)
C.	EPI-REZ 5101/APCO 322	(Elevated-Temperature Cure)

PRELIMINARY RESIN SYSTEM EVALUATION TESTS AT CRYOGENIC TEMPERATURES

NOTES

1. 6" DIA. NOL RINGS TESTED WITH SPLIT DISC IN LN_2
2. 3 NOL RINGS MADE OF EACH RESIN SYSTEM – TOTAL OF 30 RINGS
3. GLASS – E/HTS



RESIN SYSTEM	EP1 –REZ 5085 EPICURE 855	EP1 REZ 5 085 502 EPICURE 855	NARMCO 7343 7139 (MEK)	EP – REZ 510 5042 EPICURE 841	ERLA 2772 ZZL 0822	ERLB 0510 ZZL 0803	DER 332 APCO 322	EP1 – REZ 5101 APCO 322	ER X 36 CL	ERLA 2556 Z7LB 0820
POT LIFE	PASSABLE	PASSABLE	PASSABLE	PASSABLE	BAD (1 HOUR)	GOOD	GOOD	GOOD	GOOD	GOOD
TYPE CURE	ROOM TEMP.	ROOM TEMP.	ROOM TEMP.	ROOM TEMP.	ROOM TEMP	ROOM TEMP.	ELEVATED TEMP.	ELEVATED TEMP.	ELEVATED TEMP.	ELEVATED TEMP.

FIGURE 2-51

The resin system used in the Douglas preliminary program (Appendix A, Epon 828/EPICURE 855) is not suitable for larger vessels because of its short pot life.

2.2.2 Uniaxial Cyclic Tests at Cryogenic Temperatures

This work has been completed. The task consisted of the following: (1) two plies of single-end glass, S and Z twist are wound on a flat mandrel at 10 degrees each and six plies at 90 degrees; all at 100 ends per inch, (2) the laminates are then vacuum bagged and cured for the required time and at the required temperature, (3) four specimens, 1" wide by 8" long are cut from one side of the laminate (thermal contraction specimens are cut from the other side). Then, the specimens are machined to "dogbone" shape.

Since the liquid hydrogen laboratory was scheduled with high priority work and an approximate answer was wanted for fabricating the 7-1/2" diameter specimens, it was decided to run preliminary tests in LN_2 in the Physical Test Laboratory. The specimens were 6 inches long by 1 inch wide, "dogbone" shape, with a 1/2 x 3 inch test section. One specimen of each resin system was loaded to failure. Results were:

Resin System A at 1970 pounds
Resin System B at 2740 pounds
Resin System C at 1560 pounds

There was some difficulty at first in securing the specimens in the jaws, but rearrangement of the lead foil on the ends of the specimens finally resulted in breaking the specimens. Because of the scatter of the three samples, it was decided to start the cyclic testing at a 1200-pound load, which represented approximately 60% of the average failure loads. The test results are shown in Figures 2.52 to 2.54. Resin System B appeared best from these tests.

Subsequently, availability of the Cryogenic Annex made it possible to perform the cyclic tests in liquid hydrogen (Reference 47). The dog-bone specimens conformed to ASTM Standard D638-61T except that the overall length was 8 inches instead of 8-1/2" inches. Two specimens of each resin

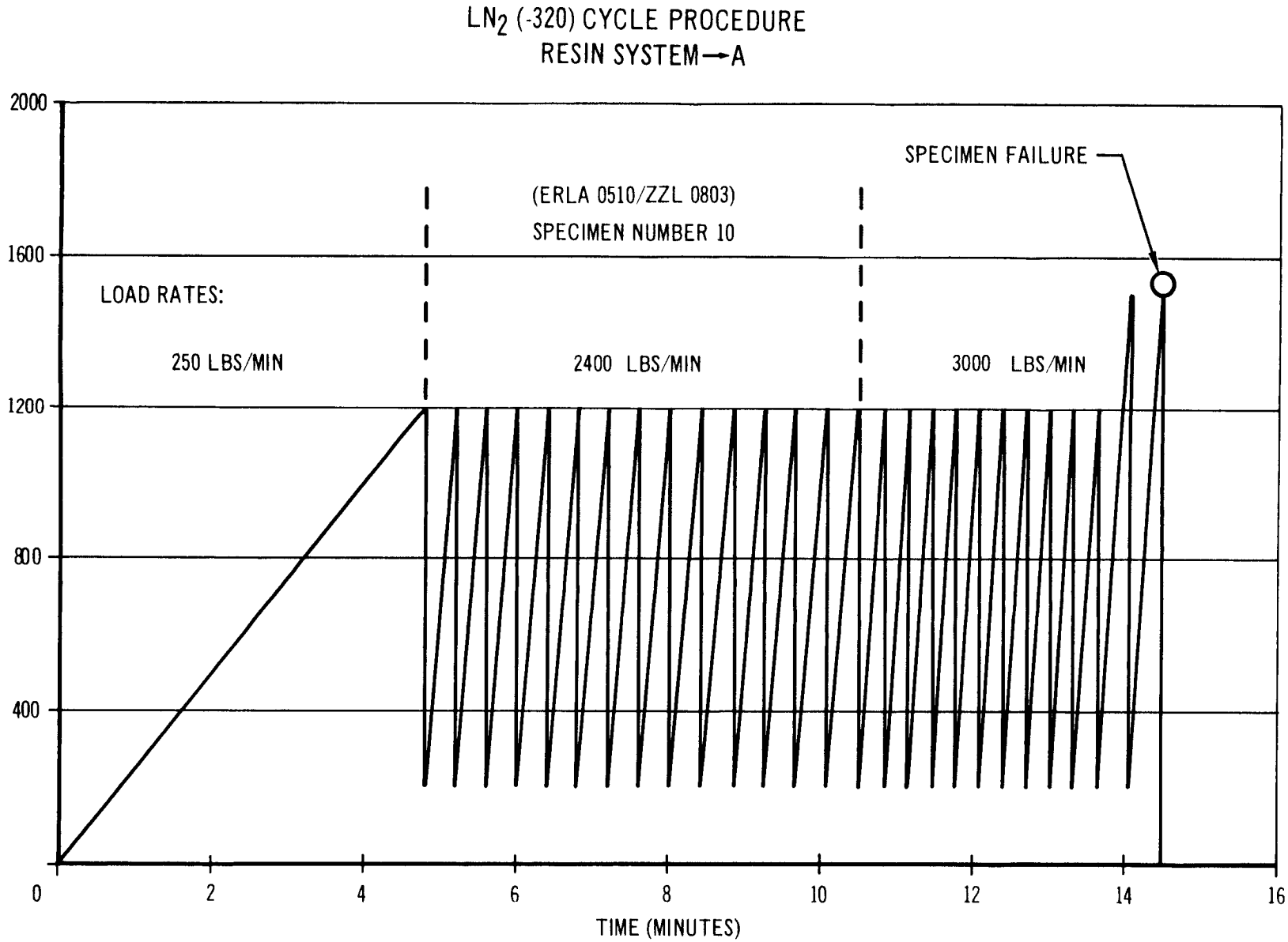

LN_2 (-320) CYCLE PROCEDURE
RESIN SYSTEM→A
2000
1600
1200
800
400
0
2
4
6
8
10
12
14
16
LOAD (POUNDS)
TIME (MINUTES)
SPECIMEN FAILURE
(ERLA 0510/ZZL 0803)
SPECIMEN NUMBER 10
LOAD RATES:
250 LBS/MIN
2400 LBS/MIN
3000 LBS/MIN

FIGURE 2-52

(EPI-REZ 510-5042/EPI CURE 841)

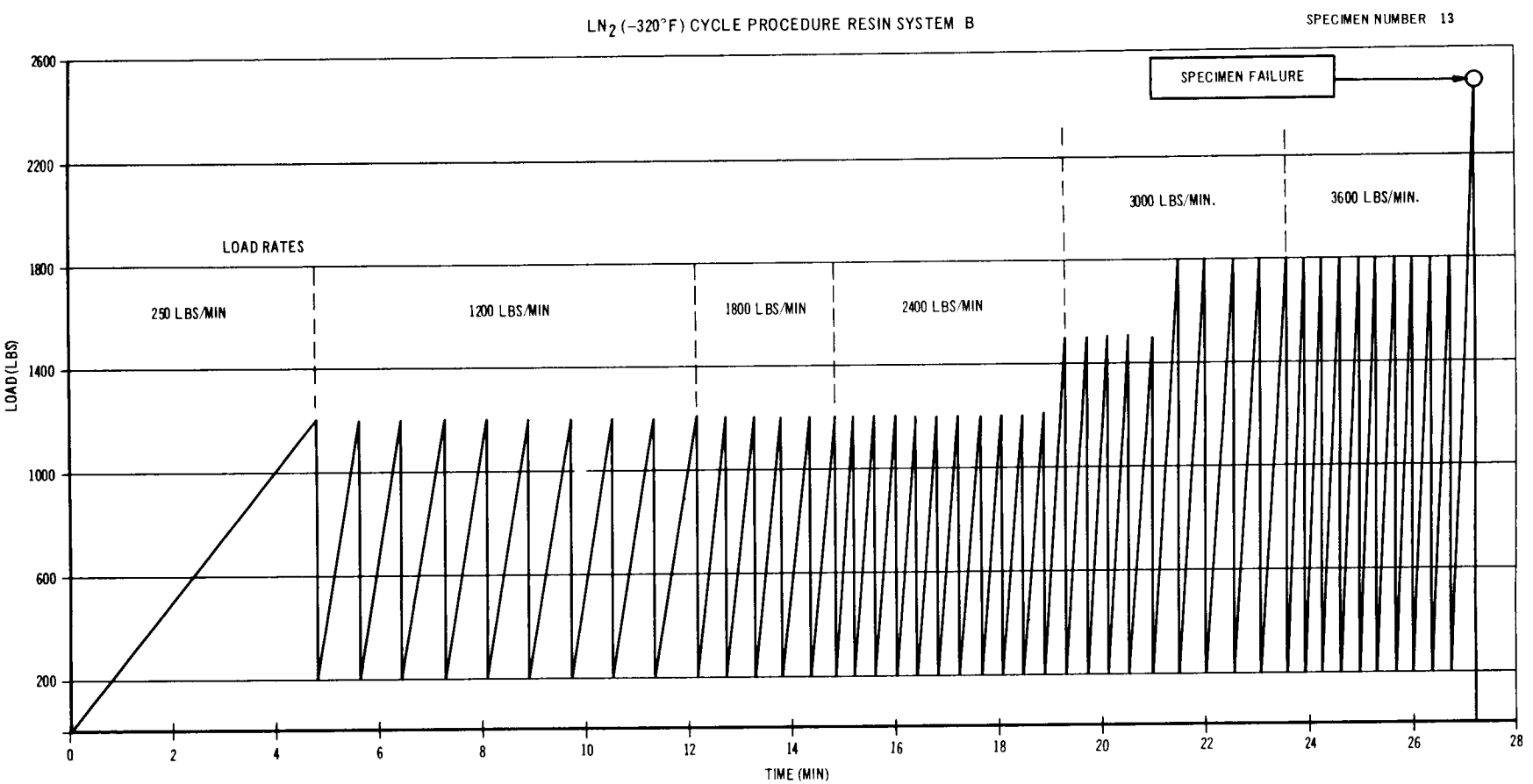


FIGURE 2-53

(EPI-REZ 5101/APCO 322)

LN_2 (-320) CYCLE PROCEDURE RESIN SYSTEM C

SPECIMEN NUMBER 16

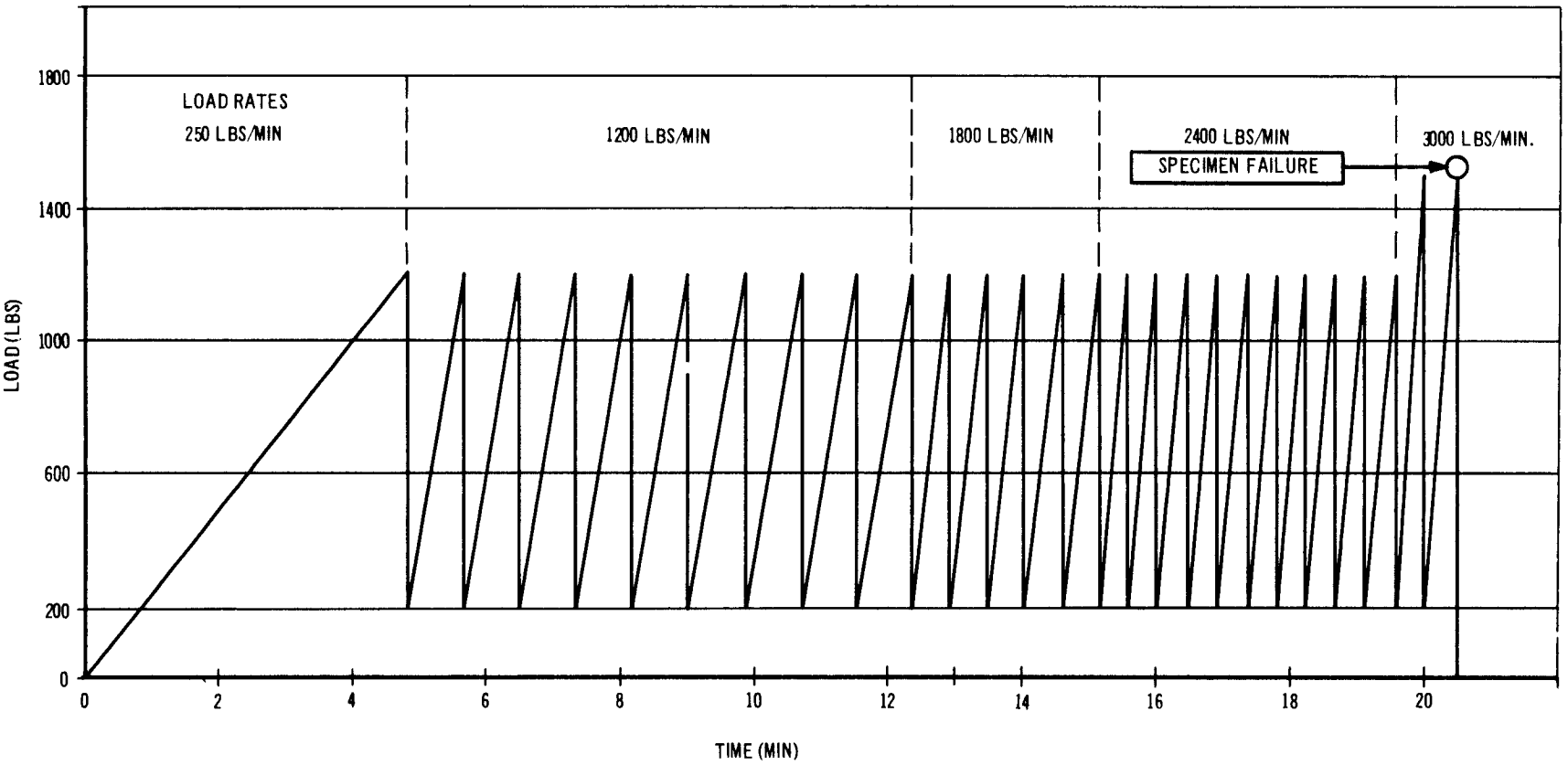


FIGURE 2-54

system were loaded to failure. Results were:

Resin System A	1265 and 1400 pounds
Resin System B	1185 and 1360 pounds
Resin System C	1740 and 2375 pounds

due to the scatter in these results and obvious non-optimum loading, it was decided to start cycling the cyclic specimens at 1200 pounds, as had been done in liquid nitrogen. The test results are shown in Figure 2-55. Resin System C appeared best from these tests. However, a room temperature curing resin system was preferred since there was no data on the effect of high temperature cure on the filins and adhesives. Therefore, Resin System A was used for subsequent biaxial fabrication in place of Resin System B.

2.2.3 Mechanical Properties

Mechanical properties tests are being made of the selected materials, using the 7-1/2-inch diameter biaxial test specimen. This specimen permits complete evaluation of the resin/fiber glass system under conditions of actual use in the vessels; i.e., resin strains and cracks in two directions.

Quality control tests revealed that all of the "S" twist single end glass roving was E-glass instead of S-994 glass, as had been ordered. As a result, the first three completed 7-1/2" diameter specimens which had been fabricated with this material were set aside for use as preliminary evaluation and check-out specimens. Fortunately, a current Douglas Program had a supply of "S" twist S-994 glass, which was made available for use in this program. Subsequent specimens have been made with both "S" and "Z" twist S-994 glass.

Tests are being performed in the vacuum test chamber. Measurements have been made for the completed burst test at ambient temperature of tensile strength, yield strength, modulus of elasticity, and ultimate elongation. The failed specimen is shown in Figures 2-56 and 2-57. Results of the test are shown in Figures 2-58 through 2-60. (Resin Content in the test section was 29.6% by weight). The ultimate hoop glass stress was high, as was expected with S994 glass.

The hoop strain of 2.32% at burst was somewhat lower than expected; longitudinal strain of 0.64% indicates an overdesign in the longitudinal

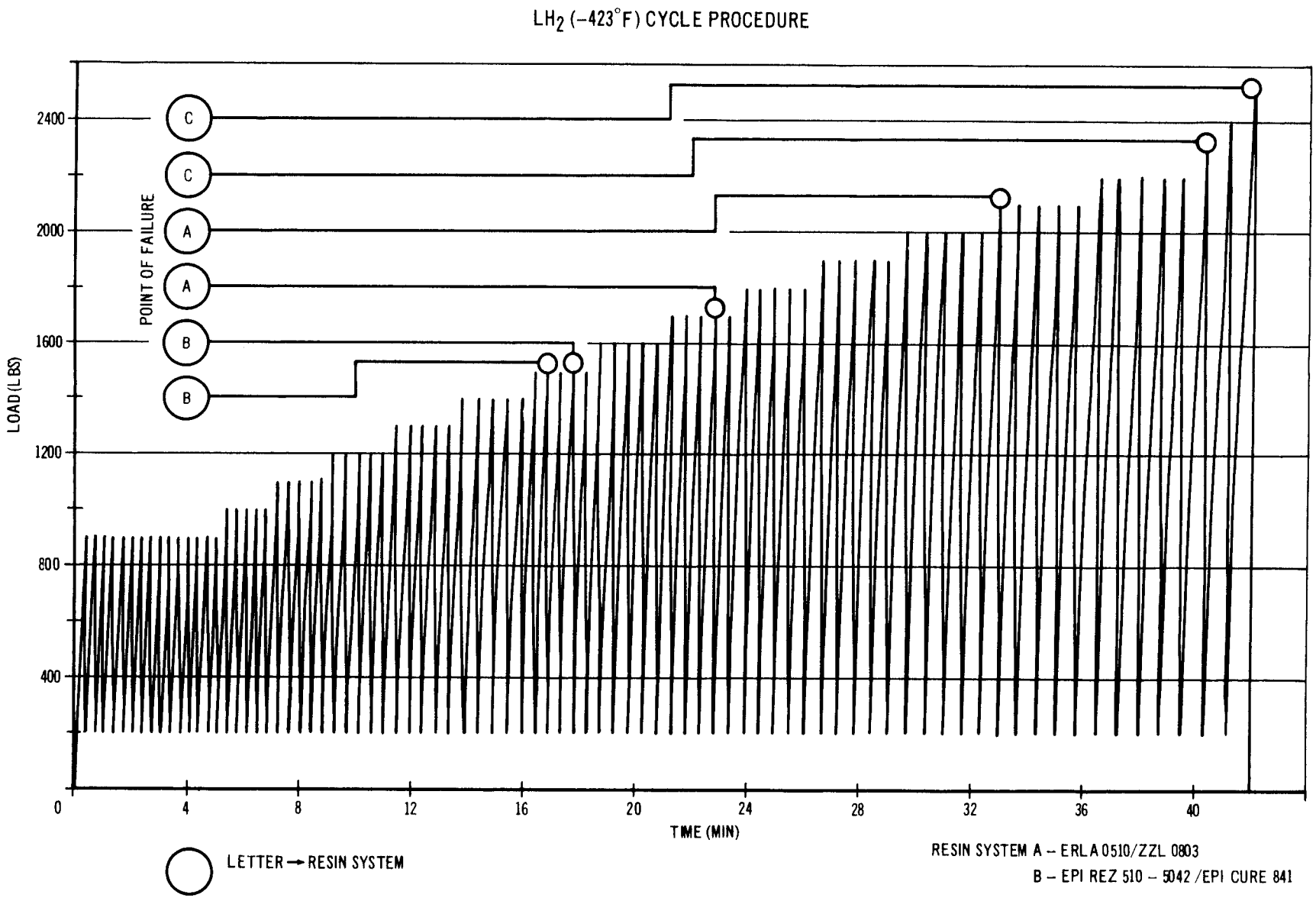

LH_2 (−423°F) CYCLE PROCEDURE
LOAD (LBS)
2400
2000
1600
1200
800
400
0
4
8
12
16
20
24
28
32
36
40
TIME (MIN)
POINT OF FAILURE
C
C
A
A
B
B
LETTER → RESIN SYSTEM
RESIN SYSTEM A – ERLA 0510/ZZL 0803
B – EPI REZ 510 – 5042 /EPI CURE 841
C – EPI REZ 5101/APCO 322

FIGURE 2-55

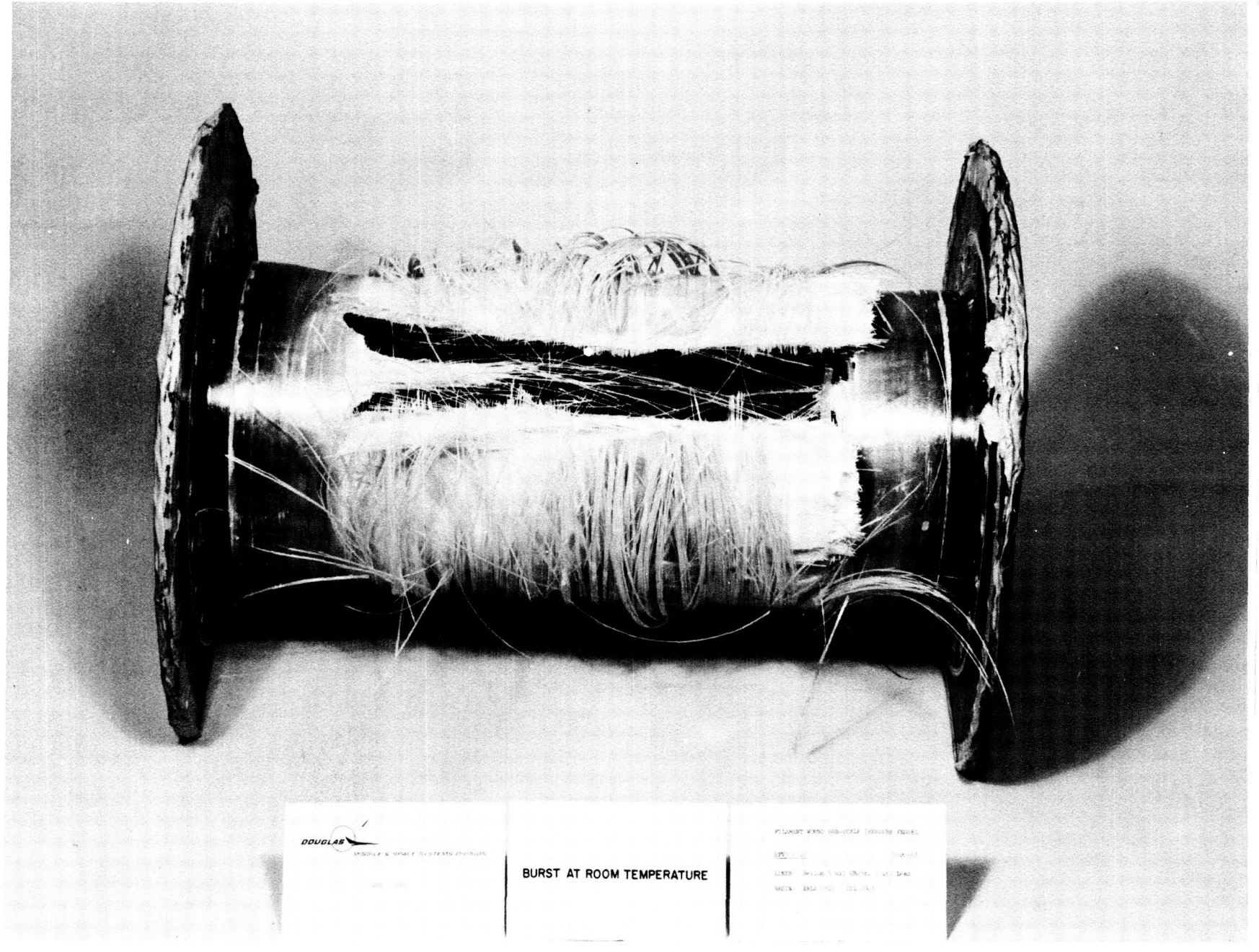
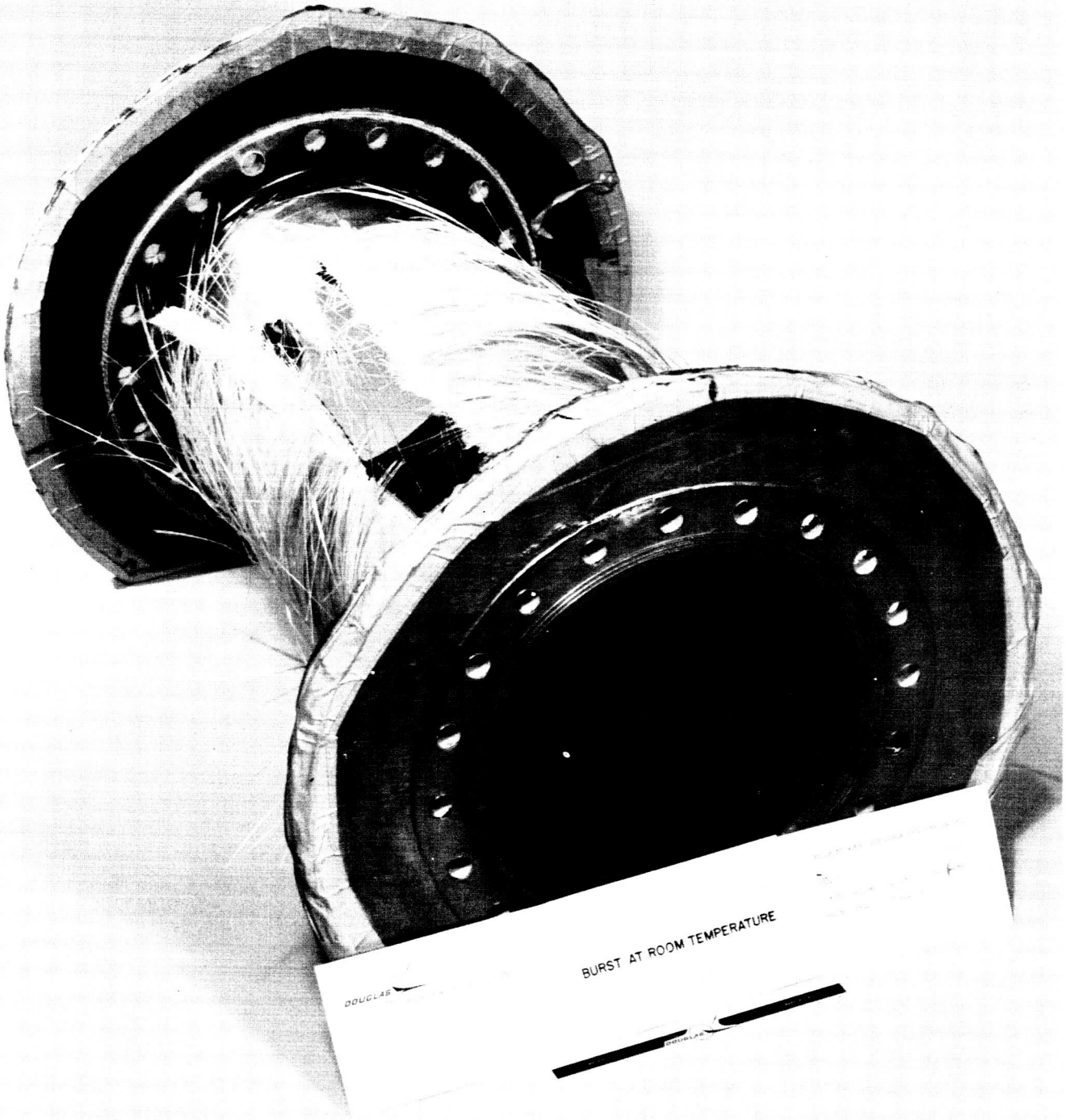


FIGURE 2-56

BURST AT ROOM TEMPERATURE



BURST AT ROOM TEMPERATURE

FIGURE 2-57

BIAXIAL TEST
PRESSURE VS TIME FOR AMBIENT TEMPERATURE BURST
SPECIMEN NO. SPV 3-10

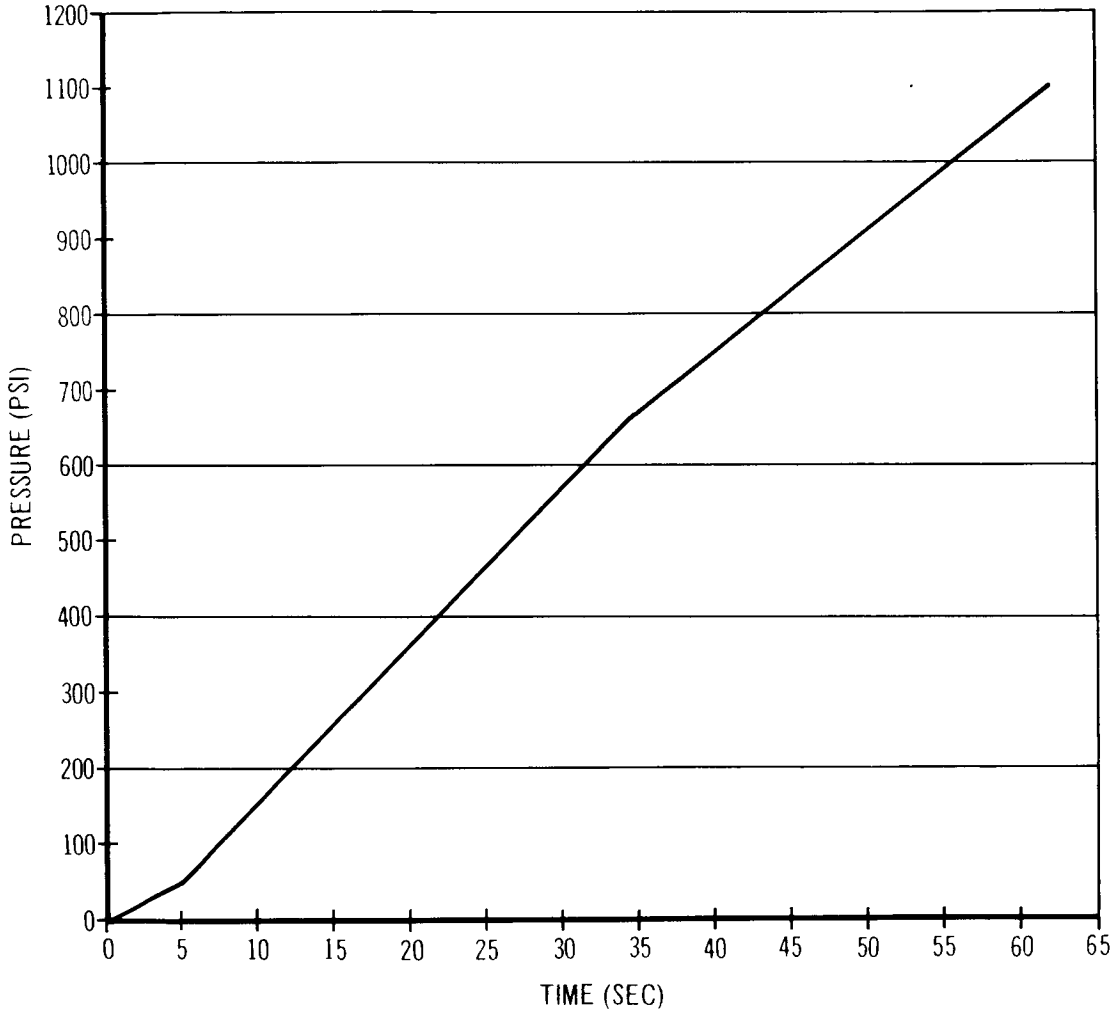


FIGURE 2-58

BIAXIAL TEST
STRESS-STRAIN DIAGRAM OF AMBIENT TEMPERATURE BURST

SPECIMEN NO. SPV 3-10

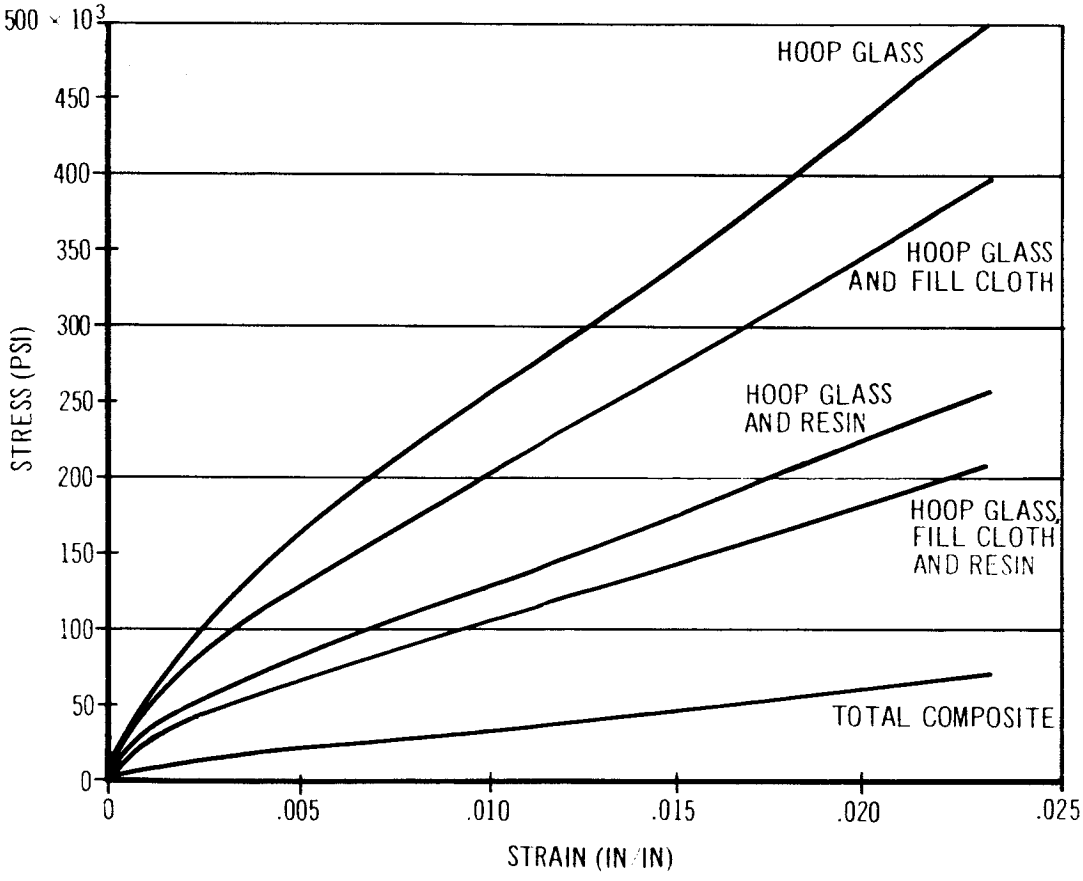


FIGURE 2-59

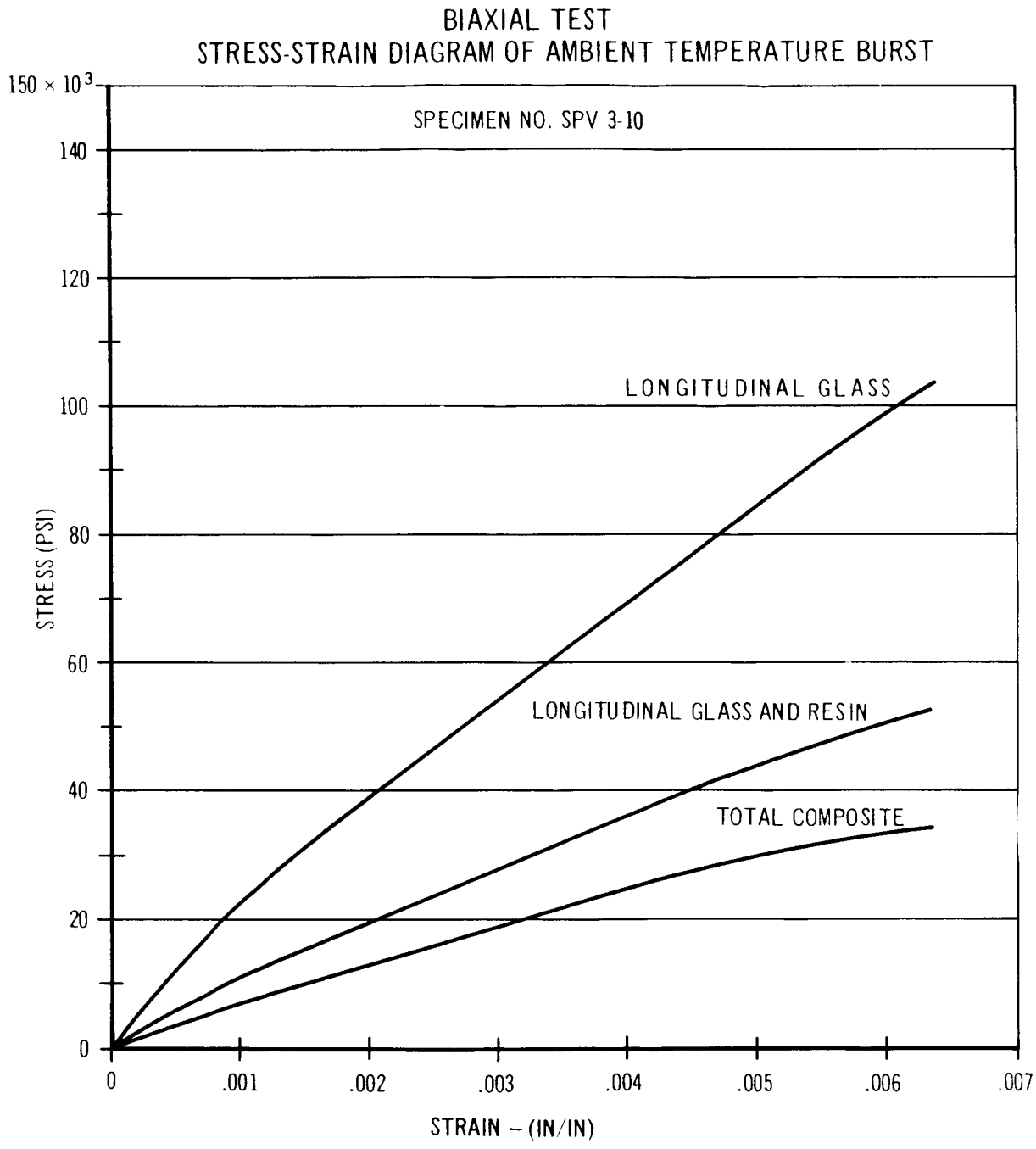

BIAXIAL TEST
STRESS-STRAIN DIAGRAM OF AMBIENT TEMPERATURE BURST
SPECIMEN NO. SPV 3-10
LONGITUDINAL GLASS
LONGITUDINAL GLASS AND RESIN
TOTAL COMPOSITE
STRESS (PSI)
150 × 10³
140
120
100
80
60
40
20
0
0
.001
.002
.003
.004
.005
.006
.007
STRAIN – (IN/IN)

FIGURE 2-60

direction. However, a sufficient number of specimens have been fabricated to cover the initial ambient temperature and liquid hydrogen work; the design will be changed if deemed necessary to cover further test work. The present design will be sustained until a break in the fabrication and test sequence is reached in order to provide necessary correlation between presently completed and soon to be completed specimens.

A perforated Mylar bag filled with polyethylene pellets will be used as a volume-reducing system in liquid hydrogen. In all probability, no internal volume-reducing system will be used for the tests in liquid nitrogen.

2.2.4 Coefficient of Thermal Contraction

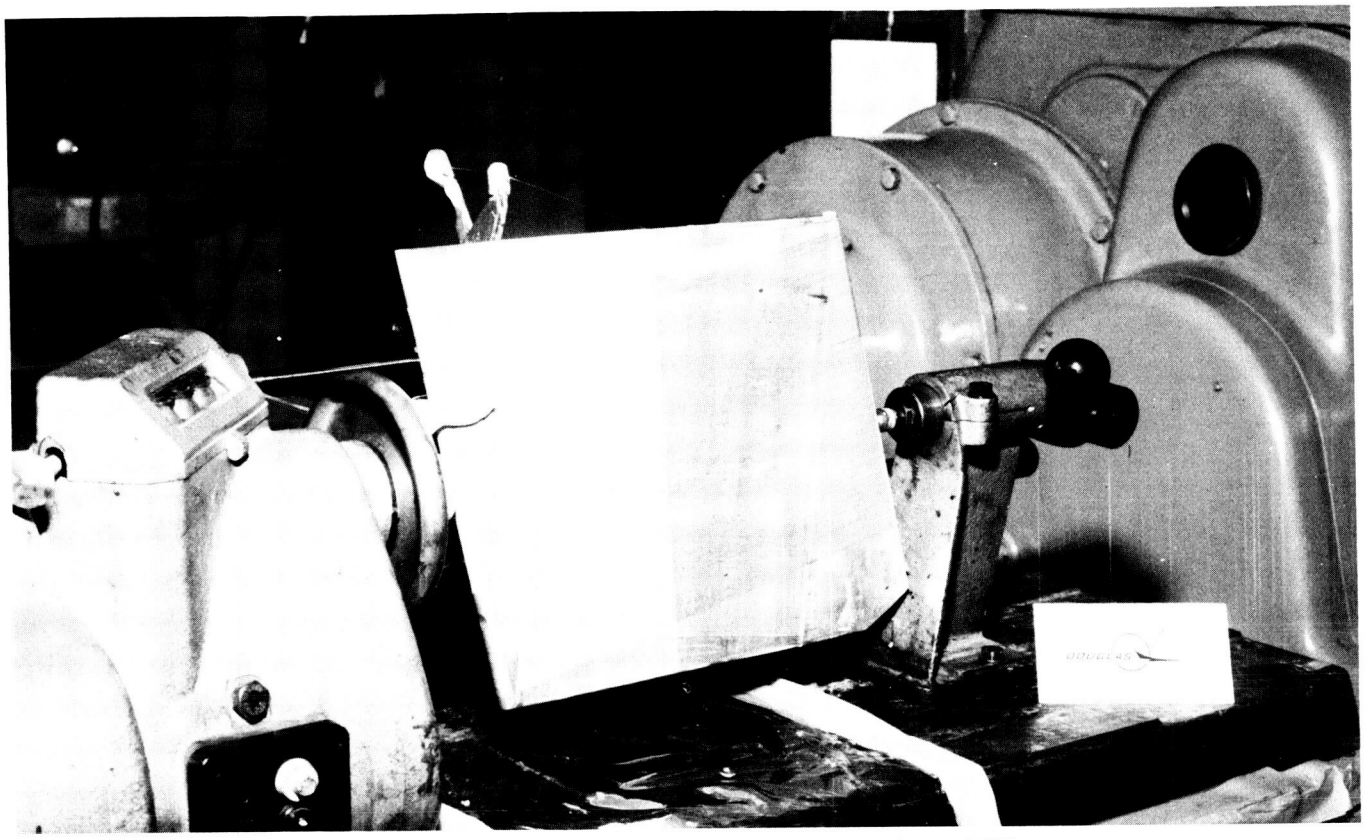
These tests were performed in the Materials Thermodynamics Laboratory and Cryogenic Annex.

In order to provide a representative specimen, simulated filament-wound laminates were made as follows: two plies of single and glass, S and Z twist were wrapped on flat mandrels at 10 degrees each and six plies were wrapped at 90 degrees with 100 ends per inch. The fabrication procedure is shown in Figure 2-61. The laminates were vacuum bagged and cured for the required time and at the required temperature. Test specimens were cut from the cured laminate and tested with the aluminum holding fixture, mentioned in Section 2.1.2, in the quartz tube dilatometer.

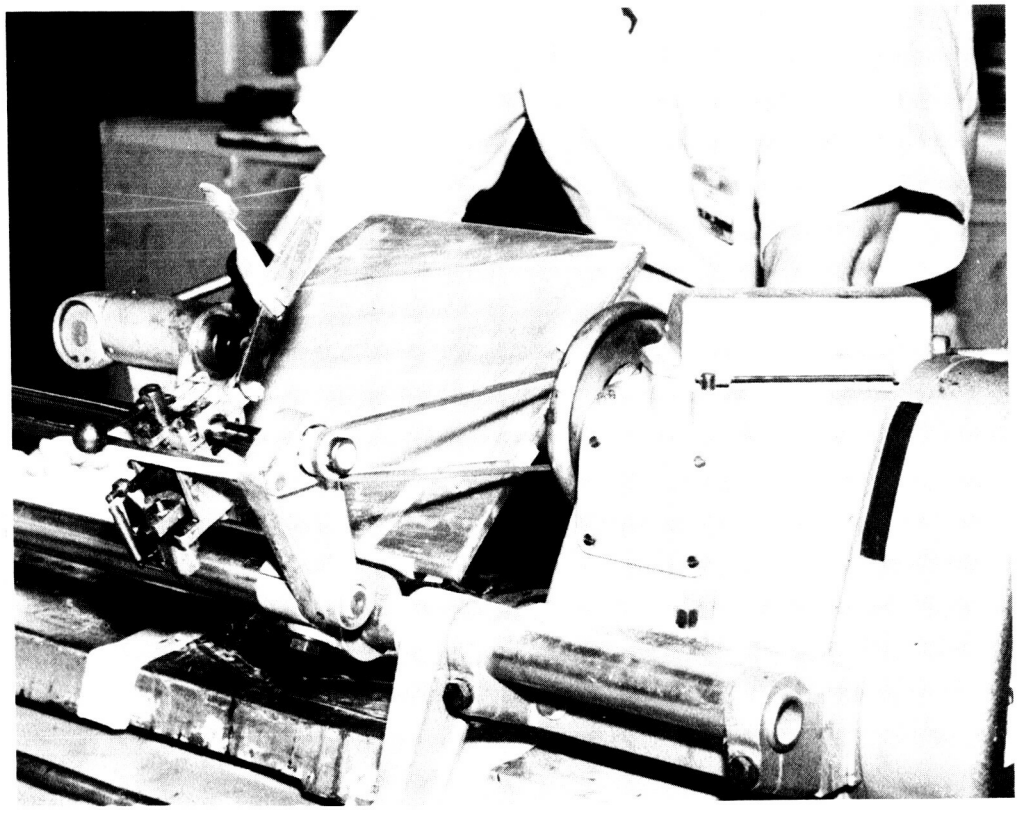
The results are included in Figure 2-39 and Appendix D. The elevated temperature cure system has a slightly lower total contraction to -423°F than the two room temperature cure systems.

2.2.5 Biaxial Cyclic Tests

The initial permeability test will be the first cycle of this series; specimens will be cycled at a rate to be determined. The specimens will be fully instrumented and cycled at 60% of the ultimate load until failure occurs. Tests will cover ambient temperature, -320°F, and -423°F work.



THERMAL CONTRACTION SPECIMEN LAMINATE
OFFSET WRAP SIMULATING TOTAL HELICAL ANGLE OF CYLINDER WALL



THERMAL CONTRACTION SPECIMEN SHEET IN FINAL
WRAP POSITION SIMULATING HOOP FILAMENT WINDING

FIGURE 2-61

2.3 VESSEL DESIGN AND FABRICATION

The design and fabrication of the two 18-inch diameter by 24-inch long fiber glass filament-wound pressure vessels are discussed below.

2.3.1 Analytical Considerations

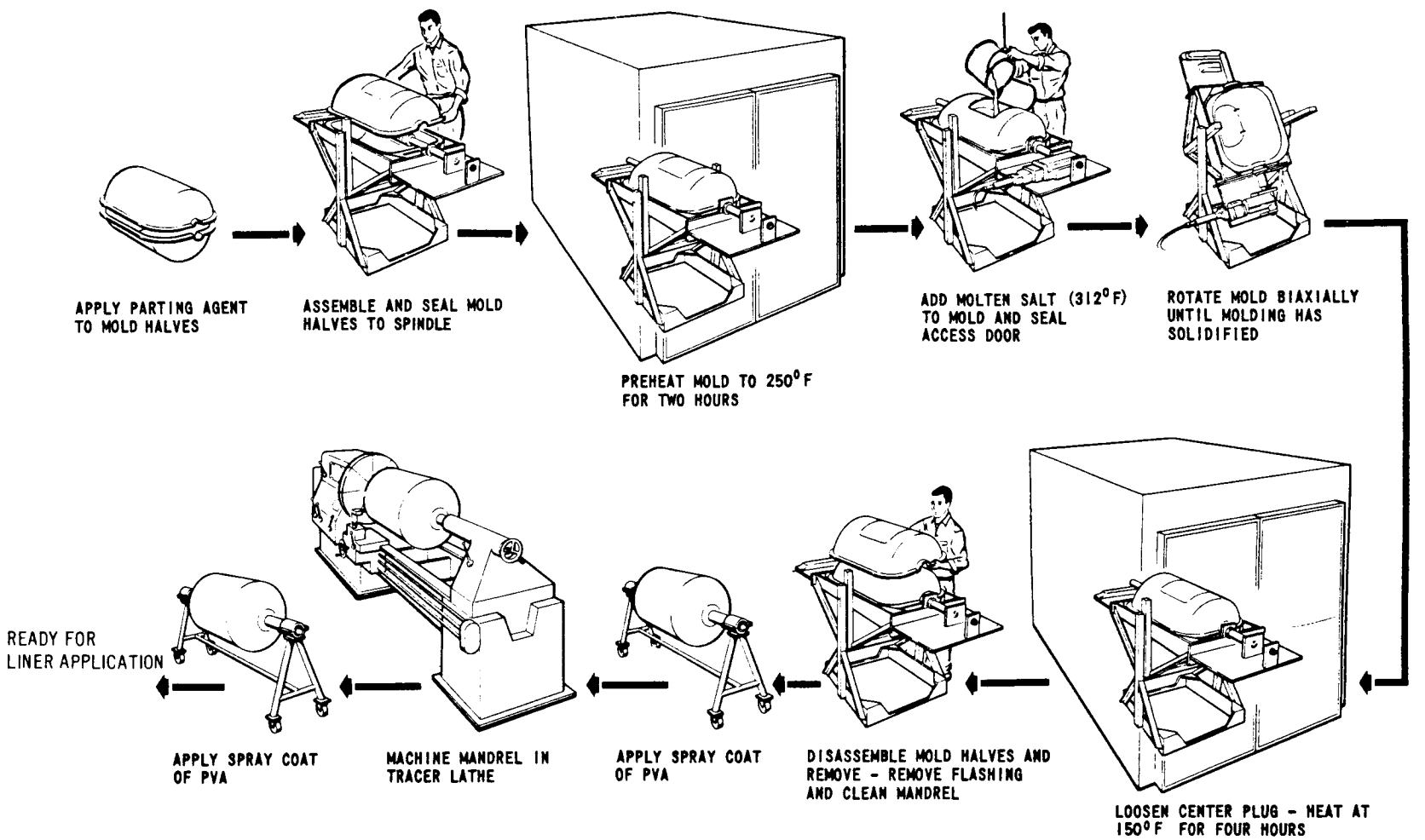
Three basic methods of filament winding have been considered in past and present Douglas work: (1) the helical wrap, (2) the sequential (polarcircumferential) wrap, and (3) the oriented longitudinal helical stave construction. The first two methods have been used in various test efforts, and the third is currently in development at Douglas.

The attainable strength-to-weight efficiency of the third method, the helical stave construction, is believed to be somewhat greater than that achieved in current filament winding; in spite of this likely advantage, the development problems associated with stave construction and with a vessel liner suitable for liquid hydrogen were not desirable for investigating in a one program effort. Because of this Douglas has directed its attention toward the already well-developed technique of helical wrapping with additional hoop wraps in the cylindrical portion of the vessel.

The basic analytical approach for helical-wound vessels is well known and has often been called the "netting analysis." This analysis ignores the resin as a load-carrying part of the vessel wall, and assumes that all load is sustained by the filaments in a membrane loading and that a small segment of selected fibers are laid parallel for each sweep direction of the filament guide. Additional analysis and experimental programs, both at Douglas and other contractors, have contributed to the design of filament-wound pressure vessels. All of the techniques are being reviewed for suitable design parameters.

2.3.2 Fabrication of Test Vessels

Soluble salt mandrels will be used in the fabrication of the two pressure vessels produced during Phase I and the 20 additional vessels produced in Phase II. The manufacturing sequence to be used is shown in Figure 2-62. The proposed filament winding sequence is illustrated in Figure 2-63.



SOLUBLE SALT MANDREL
FABRICATION SEQUENCE

FIGURE 2-6.2

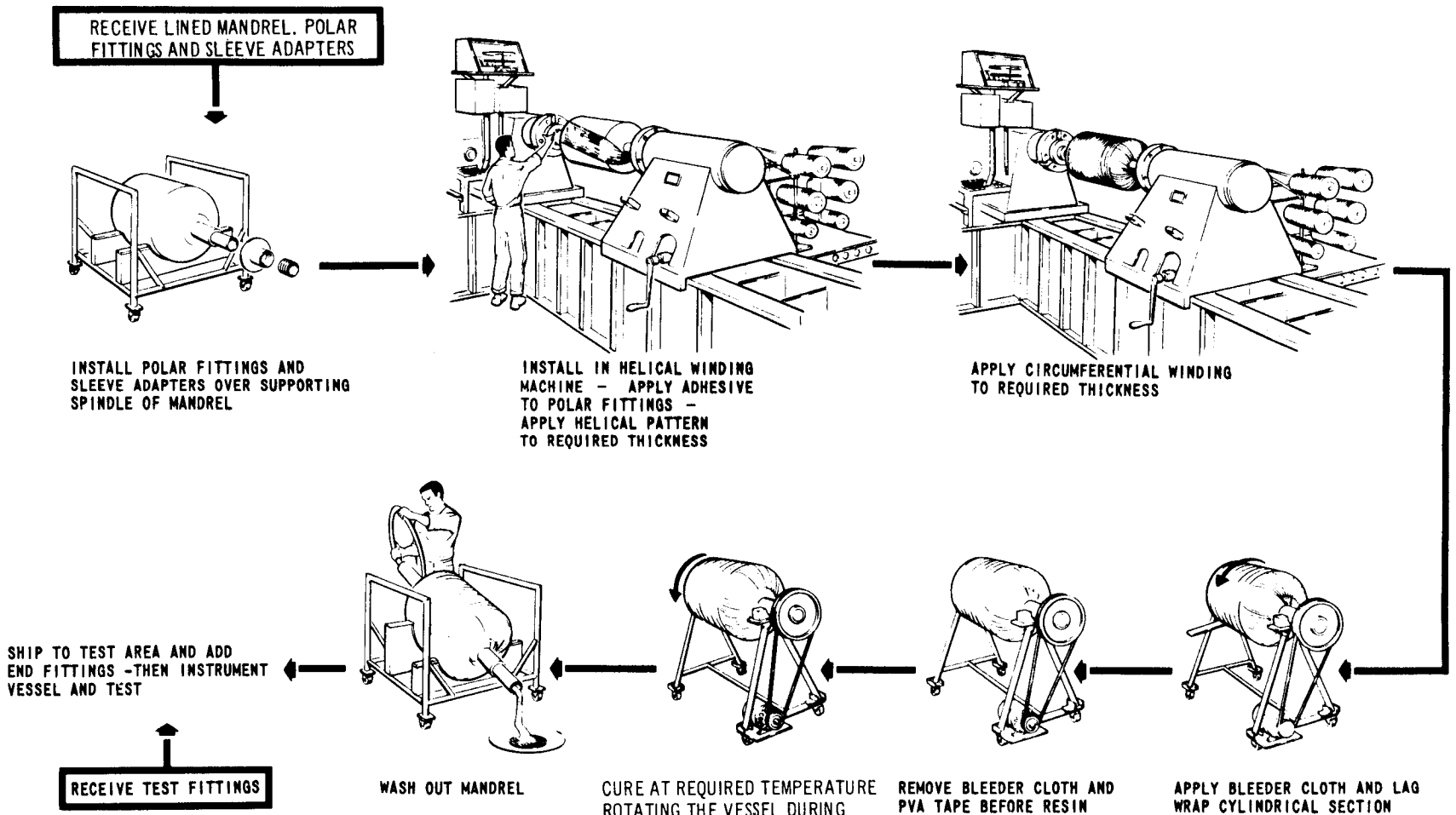


FIGURE 2-63

PRESSURE VESSEL
FILAMENT WINDING SEQUENCE

SECTION 3

PHASE II - FABRICATION OF SMALL-SCALE PRESSURE VESSELS

The objective of Phase II is to fabricate 20 small-scale fiber glass filament-wound pressure vessels for cyclic and burst testing at -320°F and -423°F. The tanks will be designed for a nominal proof pressure of 500 psi and a nominal burst pressure of 600 psi. To provide the desired mode of failure, the design may possibly have to be revised during the fabrication period; therefore, the fabrication is set up on an intermittent production schedule.

SECTION 4

PHASE III - STRENGTH AND CYCLING TESTS AT -320°F

The objective of Phase III is to determine the ultimate strength and cycling capability of 10 small-scale fiber glass filament-wound vessels at liquid nitrogen temperature. In any pressure vessel design, it is necessary to know the maximum pressure the vessel can withstand and how many cyclic loadings can be applied at a given stress level. Ultimate pressure capability is self-explanatory. Cycling tests are important in any evaluation of filament-wound pressure vessels with liners because differences in strain characteristics of the resin/fiber glass system and the liner material may cause ultimate failure of the system.

Girth and longitudinal strain measurements are necessary for evaluating the tank and liner during cycling tests and for collecting meaningful data. Of equal importance is the measurement of gas content into the burst chamber during the tests, because this measurement will be an indication of liner breakdown under cyclic loads.

Phases III and IV of the proposed program call for testing the 18-inch diameter x 24-inch long vessels manufactured in Phase II. Liquid nitrogen is to be used as the pressurizing fluid in Phase III, while liquid hydrogen is used in Phase IV; other than this, the two phases are identical in proof pressures, pressure cycling requirements, and data to be obtained.

SECTION 5

PHASE IV - STRENGTH AND CYCLING TESTS AT -423°F

The objective of Phase IV is to determine the effect of -423°F on the ultimate strength and cycling capability of 10 small-scale filament-wound fiber glass tanks. The data sought here is the same as in Phase III.

As stated in Section 4, the only difference between the test procedure of Phase III and Phase IV is that Phase IV uses liquid hydrogen rather than liquid nitrogen. The test set-up and instrumentation will be used without any changes.

SECTION 6

PROGRAM PLAN

6.1 Program Scope

Douglas is performing an investigation of the structural properties of fiber glass filament-wound pressure vessels for cryogenic fluids. The total work is in four phases and is scheduled for a period of 16 months.

Phase I - Design and development of small-scale fiber glass filament-wound pressure vessels for containing cryogenic fluids.

1. The following liner materials are being evaluated:

Mylar	Nickel Plating
H-Film	Copper Plating
Tedlar	Silver Plating
Polyurethane Film	
Glass Flakes	

Tables 6-1 through 6- 3 show the number of specimens in the evaluation.

2. The following resin/fiber glass composite are being evaluated:

Glass Filament	Resin
S-994	EPI-REZ 510-5042/EPICURE 841
S-994	ERLB-0510/ZZL-0803
S-994	EPI-REZ 5101/APLO-322

Phase II - Fabrication of twenty 18-inch-diameter by 24-inch-long pressure vessels for use with either liquid nitrogen or liquid hydrogen.

Phase III - Testing of small-scale fiber glass filament-wound pressure vessels at liquid nitrogen temperature. The test sequence is shown in Table 6-4

Phase IV - Testing of small-scale fiber glass filament-wound pressure vessels at liquid hydrogen temperatures. The test sequence is shown in Table 6-4

TABLE 6-I

LINER EVALUATION TESTS

Material	Number of Tests at Room Temperature, -320°F and -423°F	
	Uniaxial Tensile Test	Coefficient of Thermal Contraction
Mylar	5	2
H-Film	5	2
Tedlar	5	2
Polyurethane Film	5	2
Glass Flakes	5	2
Nickel Plating	5	2
Copper Plating	5	2
Silver Plating	5	2

TABLE 6-2

NUMBER OF PERMEABILITY AND CYCLING TESTS

Material	Unstressed Flat-Disc Specimen (Room Temperature)		Biaxially Stressed Cylindrical Specimen*	
	N_2	H_2	H_2	LH_2 (-423°F)
Mylar	2	2	1	2
H-Film	2	2	1	2
Tedlar	2	2	1	2
Polyurethane Film	2	2	1	2
Glass Flakes	2	2	1	2
Nickel Plating	2	2	1	2
Copper Plating	2	2	1	2
Silver Plating	2	2	1	2

* Three liners will be selected for cylinder testing at -320° F (3 cylinders of each selected liner will be made).

TABLE 6-3

NUMBER OF RESIN/FIBER GLASS COMPOSITE EVALUATION TESTS

Material Resin and S994 Glass	Uniaxial Cyclic	Biaxial Tensile Burst Tests		Biaxial Cyclic		Resin Content	Thermal Contraction and Density
	-423°F	-320°F	-423°F	-320°F	-423°F		
EPI-REZ 510-5042/ Epicure 841	3	1	1	2	2	20	3
ERLA-0510/ZZL-0803	3	1	1	2	2	20	3
EPI-REZ 5101/APCO-322	3	1	1	2	2	20	3

TABLE 6-4

PRESSURE VESSEL TESTING

Test Pressure Schedule

0 to 500 psi 0 to Burst	0 to 500 psi Cycle from 0 to 60% Net*	0 to 500 psi Cycle from 0 to 70% Net**	0 to 500 psi Cycle from 0 to 80% Net**	0 to 500 psi Cycle from 0 to 90% Net**
Tests at -423° F				
1**	2			
3				
4	7			
	8			
		11		
		12		
			15	
			16	
				19
				20
Tests at -320°F				
5				
6	9			
	10			
		13		
		14		
			17	
			18	
				21
				22

* Cycling continues until burst or through 100 cycles, whichever is earlier.

** These numbers are assigned to each vessel as they are fabricated (i.e., vessel 1 is the first vessel fabricated and vessel 22 is the last).

6.2 PROGRAM SCHEDULE

The proposed program covers a period of 16 months. Figure 6-1 shows the schedule for the four phases of the program.

PROGRAM SCHEDULE

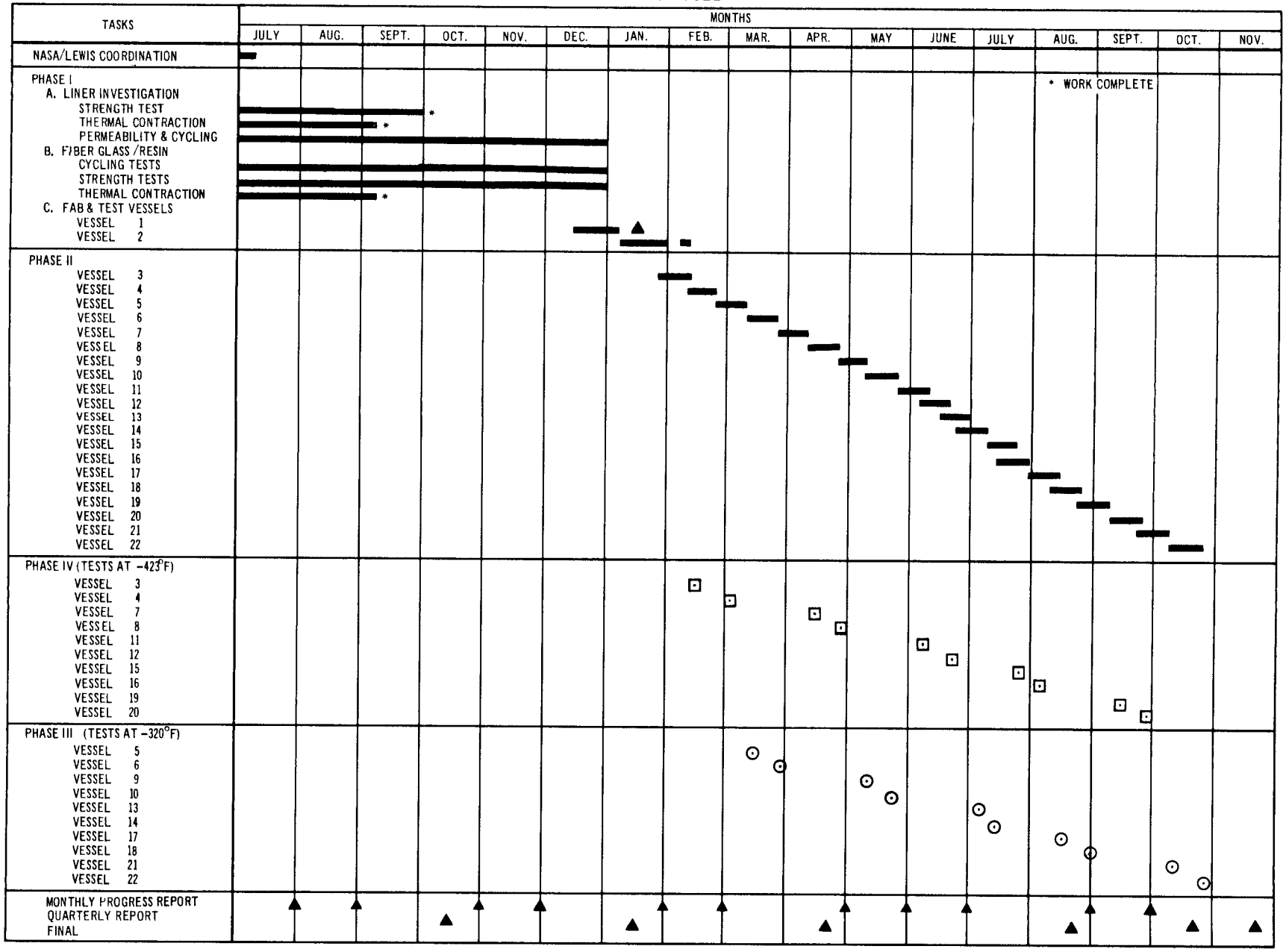


FIGURE 6-1

SECTION 7

RELIABILITY/QUALITY ASSURANCE

7.1 INTRODUCTION

The development of a research program carries with it the requirement for creating and/or adapting a quality assurance program to assure that the inherent quality of design is maintained during manufacture and test and that the subsequent test results are valid.

Interactions between the elements of the quality assurance program for design, manufacture, test, and use are defined and all phases integrated. The purpose of these efforts is to show Douglas' method of achieving acceptable function and test. Existing organization and methods of procedure are employed and will be revised as required in fulfillment of contractual obligations.

7.1.1 Organization

The Quality Assurance Program organization assures that quality requirements are adequately established and met. In recognition of this objective, the organizational structure for quality assurance provides cohesive direction, guidance, control, and assessment of an integrated Quality Assurance Program. The control agency for quality assurance is independent of the agencies whose efforts it audits and places authority at the level of management necessary to establish and enforce Quality Assurance Program policies. The keynote of the Quality Assurance Program organization is control of quality from inception of design through procurement, manufacturing, test, and use, with strong emphasis on data control.

The overall management of the Quality Assurance Program is vested in the Director of Reliability within the Missiles and Space Systems Division

(MSSD). The Director of Reliability reports to the Vice President, Director of Operations, MSSD, and directs the establishment and implementation of quality assurance functions that will assure and verify the conformance of products to contractual requirements and to Division policy relative to quality and reliability.

The functional line departments of the Reliability Director's operating department are Quality Control and Reliability Assurance. The Quality Control Department assures that design requirements are met. The Reliability Assurance Department has the responsibility for auditing the effectiveness of other departments involved in the program, the reporting of reliability/quality problems to responsible agencies, the assessment and control of data, and the effectiveness of corrective action.

7.2 DRAWING AND SPECIFICATION REVIEW

Before a designer can release a drawing, the drawing is reviewed by the appropriate group engineer, design section chief, project director, materials engineer, and schedules and planning group. These reviewers cover workability of design, dimensional and tolerance compatibility, ease of manufacture, complete material call-out, and manufacturing schedule. Before detail parts are made, the designs and specifications are further reviewed by Tooling, Manufacturing, and Quality Control in order to confirm and producibility and inspectability and to plan these operations, including the selection of characteristics to be inspected.

7.2.1 Design Release and Change Control

A fast reaction release and change control procedure is established such that the effectivity of each design change is reviewed and confirmed by the Schedules and Change Control Department, and its incorporation at the required point in production is confirmed by Quality Control. An integrated data processing system is being rapidly implemented at MSSD to

further expedite and control design release and change control functions. Some R&D design release will not require use of the full system.

7.3 DOCUMENTATION OF RAW MATERIAL TESTS

All raw material used in filament wound vessels (e.g., glass filament, resins, etc.) will be given chemical and physical laboratory acceptance tests (Figure 7-1) by Douglas in addition to those given by the supplier. Raw material received is accompanied by supplier certification reports.

Completion of the materials acceptance test requirements (M.A.T.R.) form is the responsibility of the Materials Engineer. This form is used to describe each test and test method to be employed for each material. It is used in conjunction with a laboratory work sheet. The work sheet accompanies the sample to the laboratory for testing as described in the MATR form. The Materials Engineer monitors the tests, verifies the test results, and indicates their significance with respect to value ranges given on the M.A.T.R. Material not conforming to these value ranges is rejected by Inspection and is given material review action.

7.3.1 Documentation of Test Program

Documentation of the Test Program includes test data sheets and certification of maintenance notification forms. Quality Control verification of strain gage location and installation is recorded as a permanent record and retained by the Reliability Data Center. Further test program information is given in the appropriate sections of this report.

7.4 SUPPLIER CONTROL

Since supplier furnished products are as much a part of the prime contractors design and production sequence, quality assurance control of suppliers is stressed.

ACCEPTANCE TESTS

(All Testing at R.T., 77° F)

MATERIAL	TESTS	TEST PROCEDURE
Resin	Specific Gravity	ASTM E 100-61T
	Viscosity	ASTM D 1824-61T
	Epoxy Equivalent	Special Douglas Procedure
Hardener	Viscosity	ASTM D 1824-61T
	Specific Gravity	ASTM E 100-61T
Fiber glass	Ignition Loss	OS 10782A
	Weight/Linear Yard	OS 10782A
	End Count	OS 10782A
	N.O.L. Split-Disc	Special Douglas Procedure
Plastic Film	Ultimate Tensile Strength	ASTM D 882-61T
	Ultimate Elongation	ASTM D 882-61T
	Tensile Modulus	ASTM D 882-61T
	Tear Strength	ASTM D 689-44
	Density	ASTM 1505-60T
Fiber glass Cloth	End Count Per Inch, Wrap and Fill	Special Douglas Procedure
	Temperature of Melt	Special Douglas Procedure
Adhesive	Lap Shear as Used	MIL-A-5090-D

FIGURE 7-1

Suppliers are advised of the performance of their products immediately upon determination of the performance by Douglas. If the product deviates from established requirements, the supplier is informed in writing with a request for a written corrective action statement. Corrective action statements are maintained as a permanent record for follow up and future evaluation.

7.4.1 Procurement Control

All material is procured by purchase order or contract agreement. Quality Control is responsible for adequate and effective control over procurement sources to ensure that materials, supplies, and services meet all quality requirements.

7.4.1.1 Supplier's Records

Each supplier must maintain evidence of specific tests and inspections performed and must submit a certificate of conformance. Detail test reports, verified for conformance with applicable requirements, are furnished by the supplier when:

1. The Douglas drawings or specifications require recorded data
2. The test is not witnessed by source inspection
3. The acceptance plan does not require retest at Douglas (receiving functional test).

7.4.1.2 Procurement Documents

Supplier procurement is made by purchase order with all government, engineering and quality assurance requirements indicated. The purchase order also notes whether government and/or Douglas inspection is required.

7.5 CONTROL OF DOUGLAS FABRICATED ARTICLES

7.5.1 Inspection and Test

Quality control of fabricated test specimens manufactured in Phase I of the subject program is the responsibility of the Materials Engineer. He performs sufficient initial, in-process, and final checks during the fabrication of test specimens to ensure their conformance to engineering requirements, thereby guaranteeing the validity of subsequent tests.

7.5.2 Material Control

Established procedures provide the following controls over age-sensitive raw material:

1. Complete inspection, including any required chemical or physical tests, upon receipt
2. Verification of correct marking prior to storage indicating age, date and test time as applicable
3. Verification of correct material and age requirements prior to issue for manufacture
4. Verification of correct material prior to the first fabrication operation by shop personnel.

7.5.3 Cleanliness Requirements

Cleanliness requirements are established by Design Engineering and Materials Research and Production Methods (MR&PM). Cleanliness in the production area is maintained by manufacturing under continuous surveillance by Quality Control. MR&PM controls the cleanliness in the MR&PM laboratory. Test Engineering controls the cleanliness in the Engineering R&D Laboratories.

7.5.4 Control of Nonconforming Material

Quality Control is responsible for detecting and reporting nonconforming

material. Quality Control ensures that all necessary controls are provided and implemented and that appropriate corrective action is accomplished.

7.5.4.1 Segregation

Quality Control ensures that all nonconforming material is withheld from the manufacturing system and that residual material is removed from processing operations, impounded, and identified prior to submittal to the Material Review Board.

7.5.4.2 Material Review

Quality Control submits nonconforming material to the Material Review Board for disposition. Dispositions are documented and become a permanent record for engineering evaluation.

Material Review Board (MRB): The signatures of the company Liaison Engineer and the company Quality Control Representative are required as a minimum in research and development work to signify a unanimous MRB decision. Douglas Quality Control is responsible for verifying the validity and completion of all entries and for obtaining corrective action.

Rework: When rework or repair is authorized by the MRB, Liaison Engineering records the disposition and rework instructions and references any applicable specifications. Vague or otherwise unacceptable rework dispositions are subject to nonconcurrence by Quality Control. Quality Control inspects reworked material and signifies acceptance by stamping the failure and rejection report and the accompanying manufacturing records. Quality Control submits conditional reworked material to Liaison Engineering upon completion.

Identification: If the MRB chooses to use material containing a departure from a drawing or specification (whether after rework or accepted without rework), Quality Control applies a decal to the material with the report serial number of the involved failure and rejection report, and identifies the inspection records of the next and subsequent assemblies.

7.5.5 Inspection Measuring and Test Equipment

All new test equipment, facilities, tools and gages, including masters, are inspected by Quality Control and checked-out by responsible agencies which include Product Development Support, Space Systems Engineering, Tooling, Plant Engineering, and Quality Control, as applicable. The check-out assures that all applicable specifications and requirements have been complied with and the equipment, facilities, tools, and gages used as measuring devices to determine acceptability of product are so identified by the attachment of an identification plate or certification label, which indicates the due date for recertification. Quality Control is responsible for assuring that all such devices are not used beyond the certification due date, or, if not operating satisfactorily, are withheld and identified pending repair and certification.

7.6 TEST PROCEDURES

The following procedures are used to assure that accurate and reliable results are obtained on the pressure vessel specimens.

7.6.1 Mounting of Test Specimen

The test specimen is mounted in the vacuum chamber and the associated plumbing connected. A leak test is then performed with helium leak detector unit to assure that the vacuum system is not leaking.

One copper-constantan thermocouple is attached to the test specimen.

Deflection gauges and strain gauges are mounted to measure longitudinal and circumferential expansion and strain.

7.6.2 Liquid Level Indication

A transonics model 2650-1 liquid level sensor is mounted 2 feet above the test vessel to determine when the specimen is completely filled with liquid. A light mounted in the test control panel will indicate whether the sensor is submerged or not.

A submergence test utilizing LN_2 will be conducted on the level sensor periodically to assure proper operation.

7.6.3 Pressure Measurements

7.6.3.1 Specimen Internal Pressure

The specimen internal pressure is measured with a Statham Model P10F-2MG-350 pressure transducer and recorded on a Consolidated Electrodynamics Corp. recording oscillograph. The pressure transducer and recorder are calibrated and certified to accuracy by the Douglas Metrology Laboratory.

To further increase the accuracy of the pressure transducer-recorder system a calibration of the entire system, including necessary cables, is made immediately prior to each test run. This calibration is accomplished by applying pressure in 100 psi increments to the pressure transducer while it is installed in the test setup. The reading recorded on the recorder versus the pressure applied gives a calibration of the complete system. The pressure applied is measured with a Heise test gauge (Accuracy = $\pm$ .1% of full scale).

7.6.3.2 Vacuum Chamber Pressure Measurement

The pressure in the vacuum chamber is measured with a CVC Pirani gauge. The gauge and probe are calibrated and certified as a unit by the Douglas Metrology Laboratory.

7.6.4 Temperature Measurement

One copper-constantan thermocouple will be attached to the test specimen and its output recorded on the recording oscillograph. This thermocouple when installed in the test setup will be subjected to a two point calibration utilizing LN_2 and LH_2.

7.6.5 Strain Measurement

7.6.5.1 Deflection Gauge

Six deflection gauges will be mounted on all specimens to measure longitudinal and circumferential expansion.

The output of the deflection gauges will also be recorded on the recording oscillograph. Both the recorder and deflection gauges will be calibrated and certified by the Douglas Metrology Laboratory.

7.6.5.2 Strain Gauge

Electrical strain gauges will be mounted on selected test specimens. The output of these gauges will also be recorded on the recording oscillograph.

7.6.6 Calibration

All data acquisition equipment used in the research and development test

phase of this program is standard commercial equipment. Douglas written procedures are available for this equipment and are used to calibrate each piece of equipment. Calibration certification and calibration data (e.g. calibration curves) are recorded and become permanent records.

All R&D measuring and data acquisition equipment is calibrated at periodic intervals under a computer controlled notification system. Evidence of calibration is effected by certification labels which are in accordance with current directives and are controlled by Douglas Measuring Standards.

Section 8

FACILITIES

The Douglas Missile and Space Systems Division is a large industrial and development complex devoted to the development and production of missiles and space systems.

These facilities are described below.

8.1 ENGINEERING RESEARCH AND DEVELOPMENT LABORATORIES

In Douglas' extensively equipped Engineering Research and Development Laboratories, investigations and testing functions are accomplished which include:

1. Materials tests for the determination of properties and characteristics of commercially-available and Douglas-developed metals and plastics at cryogenic and ambient temperatures.
2. Non-destructive testing of raw materials and fabricated components for purposes of inspection to assure a high degree of quality control.
3. The development of new materials, production processes for special material, or unusual application of more common material.

Propulsion Laboratory

The Propulsion Laboratory has approximately 40,000 square feet of working space equipped with a wide variety of instruments and facilities. The Laboratory has 14,000-gallon capacity for handling liquified gases and a flow rate capacity of 0 to 5,000 gpm. Supply and transfer lines are vacuum-jacketed and, in some instances, foam-jacketed with helium breather. This equipment provides the capability for a variety of experimental and developmental tests (e.g., tensile, creep, impact, fatigue, thermal conductivity, and diffusion determinations of materials at temperatures approaching absolute zero). All of the common cryogenic fluids (e.g., liquified helium, hydrogen, nitrogen, and oxygen) can be handled and employed in this facility. Incorporated in this facility is a unique high-

vacuum chamber capable of an absolute pressure of 10^{-7} mm of mercury.

A pressure test area located within the Propulsion Laboratory is available for burst testing of components with liquid hydrogen. This area has the following:

1. Two 6 x 8 x 12-foot reinforced concrete chambers.
2. Controls for cycle testing up to 5,500 psi using helium gas.
3. Pressure system to 20,000 psi for burst tests.

An over-all view of the liquid hydrogen test facility is shown in Figure 8-1.

8.2 MATERIALS RESEARCH AND PRODUCTION METHODS LABORATORIES OF AEROSPACE SYSTEMS ENGINEERING

The Douglas MR&PM Laboratories are primarily engaged in materials testing to determine the properties and characteristics of commercially available and Douglas-developed metals, plastics, and ceramics. The laboratories also engage in quality control tests, utilizing facilities for nondestructive testing of raw materials and fabricated components.

Plastics Laboratory

The Plastics Laboratory is used in initiating newly-developed plastic materials and construction into component design. Douglas has established research and testing projects in the following major fields:

1. Organic polymers and reinforcing materials
2. Design properties (physical, chemical, and functional)
3. Tooling methods
4. Fabrication methods
5. Inspection and testing techniques

The Laboratory's technical force is made up of qualified plastics engineers who conduct projects from the conception of the proposal to the static testing of the final configuration.

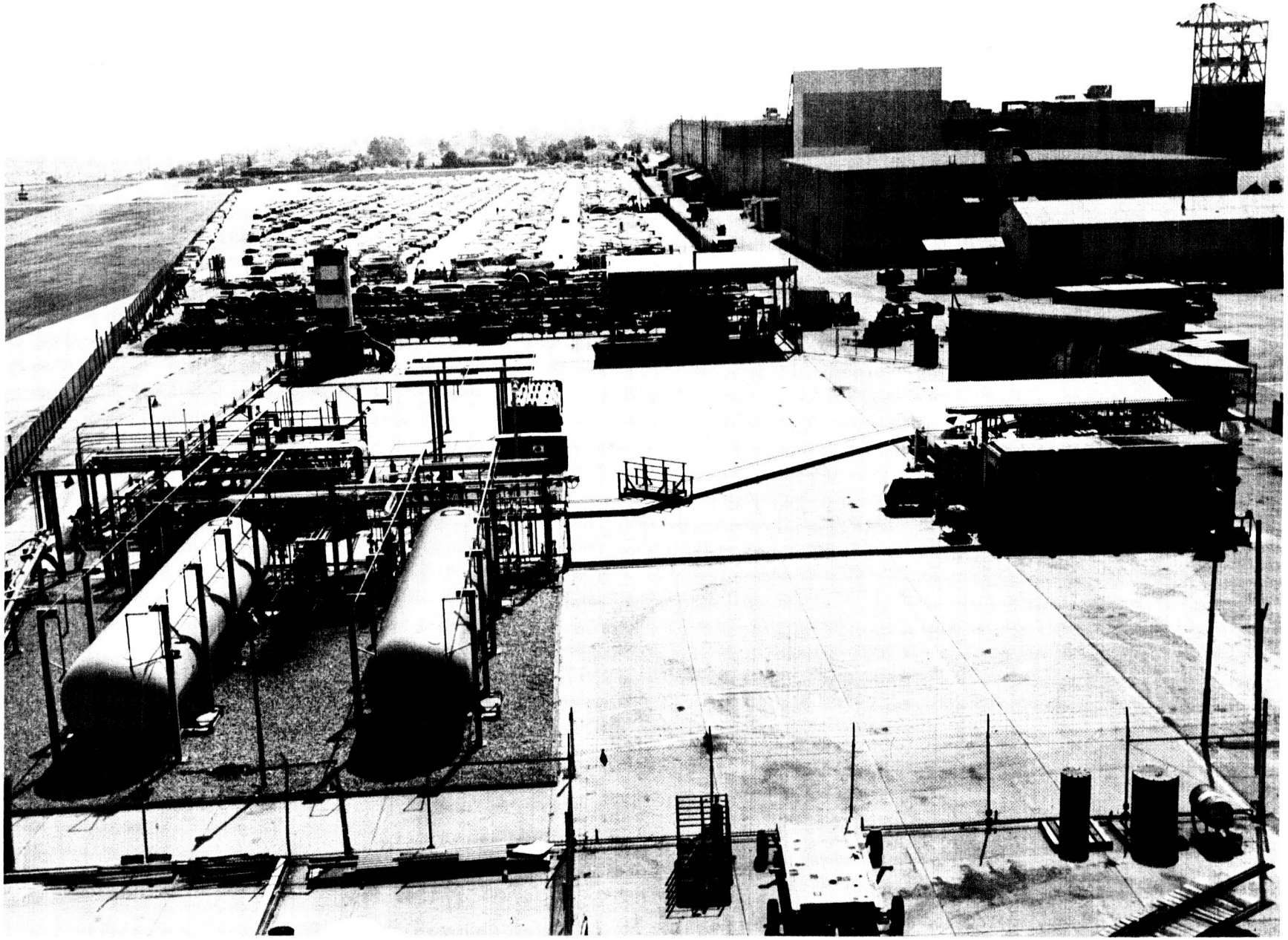


FIGURE 8-1

OVERALL VIEW OF LIQUID HYDROGEN
TEST FACILITY

The Laboratory is equipped with 3 lathe-type filament-winding machines that are capable of filament winding 2-foot-diameter by 6-foot-long cylinders. An assortment of nine circulating air ovens covering the temperature range of 100°F to 900°F is available for curing of plastic components. The Laboratory is also equipped with four heated-platen hydraulic presses, the largest of which has a 24 by 36 inch platen and a 250 ton capacity.

Equipment for testing and evaluating structural reinforced plastics and bonding (such as vacuum systems, ovens, refrigerators, and variable speed mixers) are also available.

8.2.2 Physical Test Laboratory

The Physical Test Laboratory has an area of approximately 7,600 square feet and is equipped to do mechanical properties testing of metals, plastics, and composite materials. The kinds of test that may be conducted are tension, compression, shear, bearing, flexure, creep, stress-rupture, fatigue, and impact. The Laboratory primarily contains load application equipment of three basic types: (1) universal testing machines with capacities from 5,000 to 400,000 pounds, (2) fatigue testing machines with 3,500 to 250,000 pound capacity, and (3) creep and stress-rupture equipment with capacities from 12,000 to 20,000 pounds. This equipment is supported by load strain recorders, X-Y recorders, extensometers, deflectometers, and deformation and strain indicating devices.

8.2.3 Cryogenic Annex

This is a part of the MR&PM Laboratories which is physically located in the Propulsion Laboratory. The Annex houses one 60,000-pound Baldwin tensile test machine, which is remotely controlled from a central blockhouse. Two tensile cryostats are available, one with a 10,000 pound capacity and one with a 40,000 pound capacity. Cryostat sizes can easily be changed to accommodate specimens of different configuration, depending

on the specific test requirements.

Figures 8-2 and 8-3 show the Annex and a standard tensile test in progress.

8.2.4 Materials Thermodynamics Laboratory

The Materials Thermodynamics Laboratory (Figure 8-4) has extensive equipment for a wide variety of thermophysical and thermochemical property measurements, including measurements at various temperatures of the following:

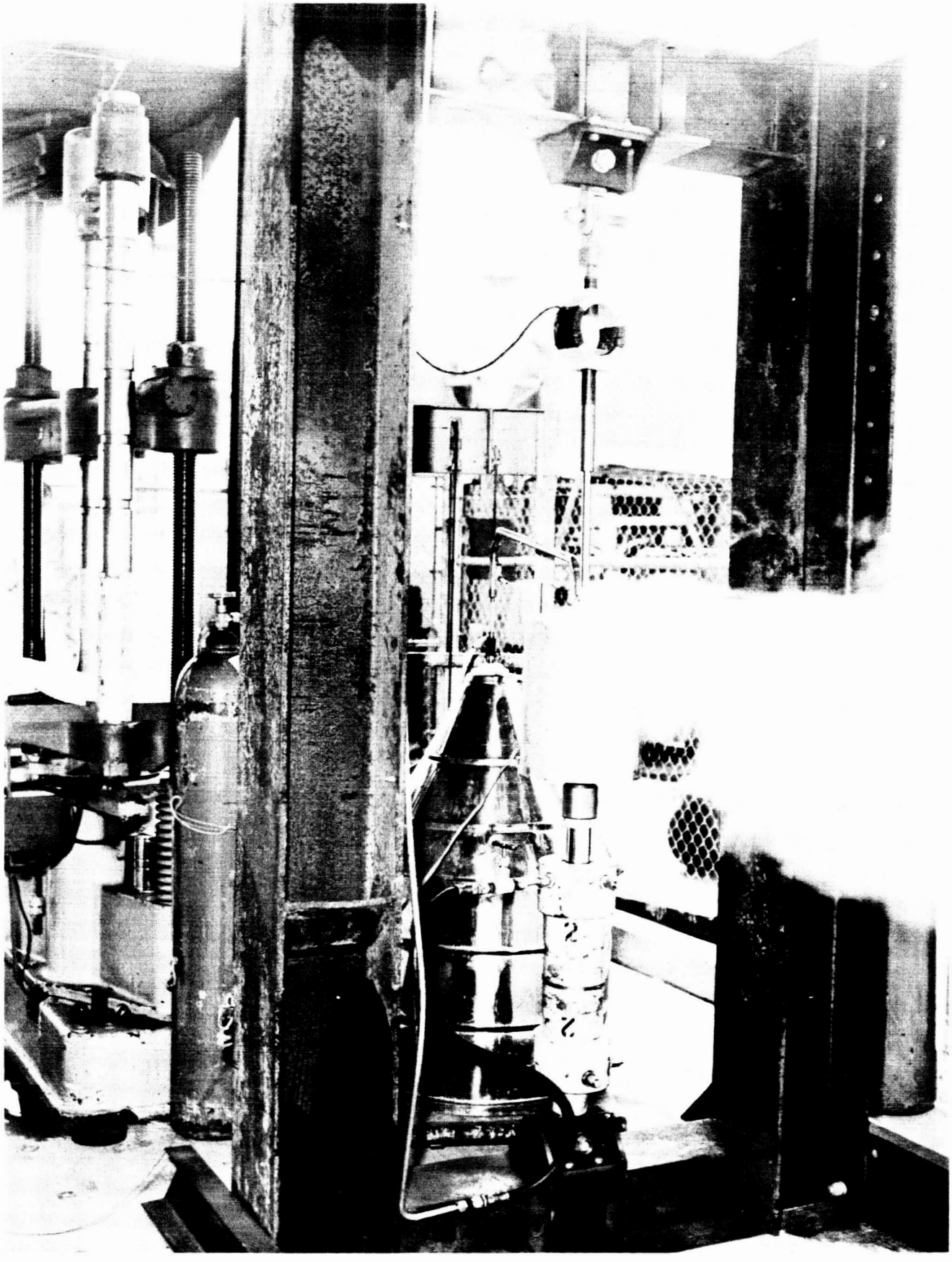
1. Thermal conductivity
2. Specific heat and heat content
3. Thermal expansion
4. Total and spectral emissivity and reflectivity
5. Solar absorptivity

The development of the equipment has stemmed from the extreme requirements of thermodynamic data for space vehicles, combat and transport vehicles, and missiles. Nearly all of the equipment has been developed and constructed in the Laboratory because of the research nature of the measurements. Many basic instruments have been acquired which are suitable and flexible for research use. A few of the items of equipment for measuring thermal properties are listed below.

1. ADL-Strong Arc Imaging Furnace with a Barnes Dual Beam (total and spectral) Radiometer
2. Modified ASTM C-177 Metal Hot Plate Set
3. ASTM C-351 Specific Heat Set
4. High Temperature Specific Heat Calorimeter
5. Quartz Tube Dilatometers Sets and heating chambers per ASTM Test Methods D95-39 and D696-44
6. ASTM C-177 Test Method Guarded Hot Plate Set
7. Resistance Thermometers, Dewars, and Recorders (for calorimetry)

CRYOGENIC ANNEX LIQUID HYDROGEN TEST LABORATORY

FIGURE 8 -2



STANDARD TENSILE TEST IN PROGRESS

FIGURE 8 -3

MATERIALS THERMODYNAMICS LABORATORY

FIGURE 8 -4

8.3 FILAMENT-WINDING MANUFACTURING EQUIPMENT

Douglas has filament-winding equipment that will wind large components up to 8-feet in diameter by 24-feet in length. These machines can be programmed to achieve special helix angles from 5° to approximately 90°. The guide eye is synchronized with the carriage motion, thus, considerable latitude is provided.

The machines are equipped with filament pre-tension devices, special resin impregnation, special tension device for packaged rovings, and resin pick-up metering arrangement and equipment.

Section 9

OUTLINE OF WORK FOR NEXT QUARTER

The work expected to be completed during the second quarter of this program is listed below:

1. All liner permeability tests (R.T., -320°F, and -423°F) will be completed.

2. All mechanical and cyclic tests of resin/fiber glass composites will be completed.

3. A liner and resin system will be chosen for the fabrication of the two 18 inch diameter by 24 inch long pressure vessels.

4. The analysis and design will be completed for the two 18 inch diameter by 24 inch long pressure vessels.

5. The tooling, shop planning, and manufacturing sequences will be started for the 18 inch diameter by 24 inch long pressure vessels.

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APPENDIX A

FIBER GLASS CRYOGENIC TANK
INDEPENDENT RESEARCH AND DEVELOPMENT PROGRAM

APPENDIX A

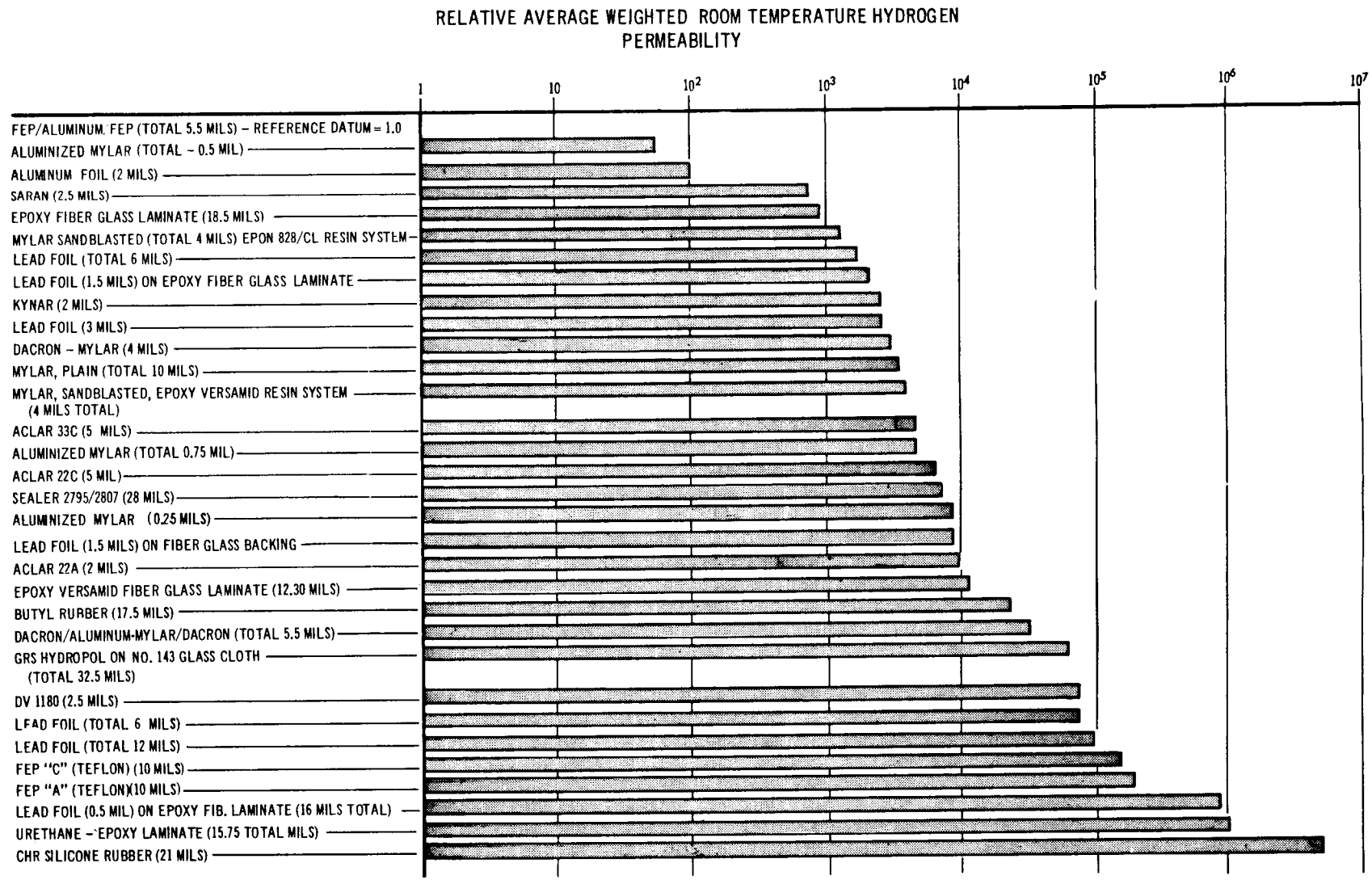
FIBER GLASS CRYOGENIC TANK INDEPENDENT RESEARCH AND DEVELOPMENT PROGRAM

An early attempt to filament-wind a bottle for containing liquid hydrogen made it clear that the selection of a suitable liner was the primary problem and that the evaluation of other desirable material and structural variables (e.g., optimum winding patterns and sandwich tank construction) would have to depend on the liner solution. Based on Douglas experience with the projected plastic Thor booster, and on the subsequent material and design research, several candidate materials were selected for evaluation. The major areas of the investigation were (1) permeability to gaseous hydrogen, (2) compatibility of unstressed samples in cryogenic fluids, (3) uniaxial strain compatibility and cycling response, (4) biaxial strain compatibility and cycling response, and (5) relative permeability in a biaxial stress field.

Room-temperature permeability tests were performed with a Douglas permeation cell on a flat-disc specimen having a slight pressure differential. Thickness of the materials tested ranged from 1/4-mil to 21 mils. The quantity of permeated hydrogen was measured with a gas chromatograph. The test results are shown in Figure A-1.

To evaluate the contraction behavior of the liner coupled to a laminate composite, a simple immersion test was performed on selected samples. The specimens were immersed in a LH_2 bath for 45 minutes; upon removal they were subjected to a qualitative examination (e.g., cracks and color change). All films tested (Saran-coated Mylar, lead foil, aluminum foil, aluminized Mylar, and Kynar) reacted favorably except the aluminized Mylar, which showed cracks upon flexing, and Kynar, which was very brittle and cracked without external load.

Strain compatibility tests were made with tensile specimens in LN_2 and LH_2. The liner materials were bonded to the fiber glass composite and



ALUMINUM FOIL (TOTAL-2MILS) – NO MEASURED PERMEABILITY

ALUMINUM/MYLAR/ALUMINUM (2.4 TOTAL MILS) – NO MEASURED PERMEABILITY

FM 1000/ALUMINUM/FM 1000 (19 TOTAL MILS) – NO MEASURED PERMEABILITY

LEAD FOIL (3 MILS) ON EPOXY FIBER GLASS LAMINATE – NO MEASURED PERMEABILITY

FIGURE A-1

cycled at varying percentages of the ultimate loads. In LN_2 the organic films performed satisfactorily, while all of the metals showed high residual elongation and debonding. In LH_2 the Mylar performed best among the plastics, and the metals continued to show high residual elongation, debonding, and cracking. Complete results of each material tested are shown in Tables A-1 and A-2.

A special Douglas-designed flanged test specimen (Figure A-2) was used to provide the needed information on biaxial strain compatibility. Evaluation tests were performed to verify the preliminary design concepts. Flatwise compression tests proved that the flange could carry bearing pressures in excess of 50,000 psi at room temperature. Leakage tests on the end seals revealed that the design was capable of sealing internal pressures in excess of 8000 psi at LH_2 temperatures.

The first specimens tested were made with lead-foil liners, while others were made with spiral wraps of Mylar tape. These materials had reacted favorably in the preliminary screening tests, were readily available, and were relatively easy to fabricate. It was difficult to obtain ultimate strength information because of the high internal pressures which were demanded and the numerous joints in the liners. It was found that a better adhesive was needed for the Mylar and a better method of fabrication for the metal liner. Recent work with a nickel-plated specimen and a Mylar-lined specimen have yielded the best results. Preliminary work has shown that it is possible to plate the soluble salt used for mandrels in fabricating Navy qualification pressure vessel. Results of these tests are shown in Table A-3.

Newer resin/fiber glass systems have shown promise of higher strengths in uniaxial tests on 3-inch-diameter test vessels. Comparison of a number of systems is shown in Figures A-3, A-4, and A-5 (Reference 42).

TABLE A-1

LIQUID NITROGEN TENSILE TESTING *

TYPE LINER & THICKNESS	SPECIMEN	LOADING – % OF ULTIMATE			
		10%	25%	50%	75%
SAND BLASTED MYLAR (0.002")	1	BOND SLIGHTLY LOOSENED BETWEEN FILM AND LAMINATE. – WRINKLES IN FILM OTHERWISE O.K. – CRACKS IN RESIN	MORE FILM WRINKLES	CRAZED IN ONE AREA – BOND BETWEEN FILM AND LAMINATE BROKEN	SPECIMEN CRACKED AT 61% – MYLAR CRACKED
	2	SAME AS ABOVE	SAME AS ABOVE	FILM O.K. – BOND LOOSENED BETWEEN FILM AND LAMINATE – WRINKLES IN FILM IN SPOTS	FILM BECAME UNBONDED IN ONE PART – FILM CRACKED IN ANOTHER
LEAD (.003")	3	BOND BETWEEN FOIL AND LAMINATE BROKEN IN CENTER OF SPECIMEN. – LEAD FOIL O.K	BOND BETWEEN FOIL AND LAMINATE BECAME WORSE – LEAD O.K., BUT ELONGATED	LEAD BROKEN ON UNBONDED SECTION	TESTING STOPPED
	4	FOIL WRINKLED BADLY AS LEAD ELONGATED – LEAD FOIL O.K.	VERY BAD WRINKLES – BOND LOOSE OVER END	LEAD BROKEN ON UNBONDED SECTION	TESTING STOPPED
ALUMINUM/MYLAR/ALUMINUM (0.0024")	5	BOND BETWEEN FILM AND LAMINATE O.K. VERY SLIGHT WRINKLES IN FILM	SAME AS 10%	BOND BETWEEN LAYERS BROKEN – WRINKLES IN BONDED ALUMINUM LAYER	MYLAR FILM BROKEN – BONDED ALUMINUM FOIL CRACKED
	6	SAME AS ABOVE	SAME AS ABOVE	WRINKLES IN FILM BUT FILM AND ALL BONDS O.K.	BONDS BETWEEN LAYERS BROKEN – TOP ALUMINUM AND MYLAR LAYERS O.K.
FEP "C" (.005")	7	FILM AND BOND BETWEEN FILM AND LAMINATE O.K.	FILM AND BOND BETWEEN FILM AND LAMINATE O.K.	FILM AND BOND BETWEEN FILM AND LAMINATE O.K.	SPECIMEN FAILED AT 65% – FILM O.K.
	8	SAME AS ABOVE	SAME AS ABOVE	SAME AS ABOVE	FILM AND BOND BETWEEN FILM AND LAMINATE O.K.
LEAD (.0015")	9	SOME WRINKLES IN LEAD FOIL – POSSIBLE CRACKS IN FOIL	BOND LOOSENED BETWEEN FOIL AND LAMINATE – WRINKLES AND CRACKS IN FOIL.	FOIL BADLY CRACKED AND BOND BROKEN BETWEEN FOIL AND LAMINATE	TESTING STOPPED
	10	SOME WRINKLES IN FOIL – POSSIBLE CRACKS IN FOIL	BOND LOOSENED BETWEEN FOIL AND LAMINATE – WRINKLES AND CRACKS IN FOIL BUT NOT AS BAD AS ABOVE	SAME AS AT 25%	SPECIMEN FAILED AT 69%
ALUMINIZED MYLAR (.00025")	11	BOND BETWEEN FILM AND LAMINATE O.K. – CRACKS IN ALUMINUM COATING	SAME AS AT 10%	BAD CRACKS IN LAMINATE – SPECIMEN FAILED ON 5TH CYCLE	TESTING STOPPED
	12	SAME AS ABOVE	SAME AS ABOVE	CRACKS IN LAMINATE BUT NOT AS BAD AS ABOVE	MORE SMALL CRACKS
ACLAR 33C (.005")	13	FILM AND BOND BETWEEN FILM AND LAMINATE O.K. – CRACKS IN LAMINATE RESIN	FILM BECAME FULLY UNBONDED FROM LAMINATE	TESTING STOPPED	TESTING STOPPED
	14	SAME AS ABOVE	FILM BECAME FULLY UNBONDED FROM LAMINATE	TESTING STOPPED	TESTING STOPPED
KYNAR (.002")	15	FILM BECAME CRACKED AND EMBRITTLED	TESTING STOPPED	TESTING STOPPED	TESTING STOPPED
	16	BOND BROKEN BETWEEN FILM AND LAMINATE-FILM O.K.	TESTING STOPPED	TESTING STOPPED	TESTING STOPPED
SARAN-COATED MYLAR (.0015)	17	FILM AND BOND BETWEEN FILM AND LAMINATE O.K. – CRACKS IN LAMINATE RESIN	SAME AS AT 10% – SARAN SLIGHTLY WRINKLED	SOME SMALL CRACKS IN SARAN	SPECIMEN FAILED AT 70%
	18	SAME AS ABOVE	SAME AS ABOVE	SAME AS ABOVE	SAME AS ABOVE

* LINER SPECIMEN BONDED TO FIBER GLASS LAMINATE.

TABLE A-2
LIQUID HYDROGEN TENSILE TESTING*

TYPE & THICKNESS	SPECIMEN	LOADING FIVE CYCLES AT 50% OF ULTIMATE
LEAD (.003")	1	FOIL WRINKLED – BOND BETWEEN FOIL AND LAMINATE BROKEN. TESTING MACHINE JAWS CUT FOIL AT ENDS OF SPECIMEN
	2	SAME AFTER CYCLING TWO TIMES AT 45% OF ULTIMATE.
STAINLESS STEEL (.002")	3	BOND BETWEEN FOIL AND LAMINATE BROKEN – FOIL ELONGATED ONE END OF FOIL CUT BY MACHINE JAWS UPON REMOVAL FROM MACHINE.
	4	BOND BROKEN BETWEEN FOIL AND LAMINATE – FOIL ELONGATED
BERYLLIUM COPPER (.002")	5	BOND BROKEN BETWEEN FOIL AND LAMINATE – FOIL ELONGATED
	6	BOND BROKEN BETWEEN FOIL AND LAMINATE – FOIL ELONGATED – FOIL CUT BY MACHINE JAWS UPON REMOVAL FROM TESTING MACHINE.
COPPER (.001")	7	(LEAD CUSHION USED ON ENDS OF SPECIMEN) BOND BROKEN BETWEEN FOIL AND LAMINATE – ELONGATION AND WRINKLES IN FOIL.
	8	SAME – TESTING MACHINE JAWS CUT ONE END OF FOIL.
NICKEL (.002")	9	(LEAD CUSHION USED ON ENDS) BOND BROKEN BETWEEN FOIL AND LAMINATE – FOIL ELONGATED – TESTING MACHINE JAWS CUT ONE END OF FOIL.
	10	SAME EXCEPT FOR NO CUTTING OF FOIL BY TESTING MACHINE JAWS
SAND BLASTED MYLAR (.002")	11	FILM WRINKLED SLIGHTLY, BUT NOT CRACKED OR BROKEN.
	12	SAME
FEP "C" (TEFLON) (.005")	13	FILM CRACKED IN MANY SPOTS
	14	SAME
ACLAR "33C" (.005")	15	BOND BROKEN BETWEEN FILM AND LAMINATE – FILM O.K.
	16	FILM VERY BADLY CRACKED
ALUMINUM (.0015")	17	FOIL CRACKED
	18	SAME
LEAD (.003") WITH NARMCO 3170	19	FOIL WRINKLED AND CRACKED
	20	SAME

* LINER SPECIMEN BONDED TO FIBER GLASS LAMINATE.

3-IN-DIAMETER CYLINDER FOR BIAXIAL STRAIN COMPATABILITY TESTS

FIGURE A - 2

TABLE A-3

BI-AXIAL TESTING CONDITIONS AND STRENGTHS

SPECIMEN	LINER AND LINER THICKNESS (MILS)	ENVIRON-MENT	PRESSURIZ-ING MEDIUM	INTERNAL PRESSURE (PSI)	HOOP STRESS (Ksi) ***	
					GLASS STRENGTH	COMPOSITE * STRENGTH
1	LEAD (3)	LH_2	HELIUM	1100	130.1	**
2	LEAD (3)	LN_2 AIR	N_2 GAS WATER	– 2700	– 330.6	– 160.1
3	MYLAR (4)	LN_2 LH_2 WATER AIR	N_2 GAS HELIUM HELIUM WATER	– 70 600 1900	– – 60.7 217.4	– – ** **
4	MYLAR (4)	LH_2 LH_2	HELIUM HELIUM	900 2000	96.9 229.4	50.2 217.8
5	MYLAR (8)	LH_2	HELIUM	1250	127.2	**
6	LEAD (12)	DESTROYED DURING REMOVAL OF MANDREL				
7	LEAD (12)	LH_2	HELIUM	3160	371.4	199.4
8	MYLAR (10)	LH_2	HELIUM	2250	243.5	119.5
9	MYLAR (10)	LN_2	LN_2	1520	158.5	85.6
10 ⊕	NICKEL (5)	LH_2	HELIUM	1) 3960 2) 4100	444.0 471.0	256.0 272.0
11 +	MYLAR (6)	LH_2	HELIUM	2100	472.0	270.0

*COMPOSITE STRESS DETERMINED FROM ACTUAL TEST SPECIMEN. NO EFFORT MADE TO KEEP RESIN CONTENT DOWN.

**RESIN CONTENT NOT AVAILABLE.

***LINER STRENGTH SUBTRACTED FROM GROSS WALL STRENGTH

⊕ CYCLED ONCE

+ REDUCED WALL THICKNESS

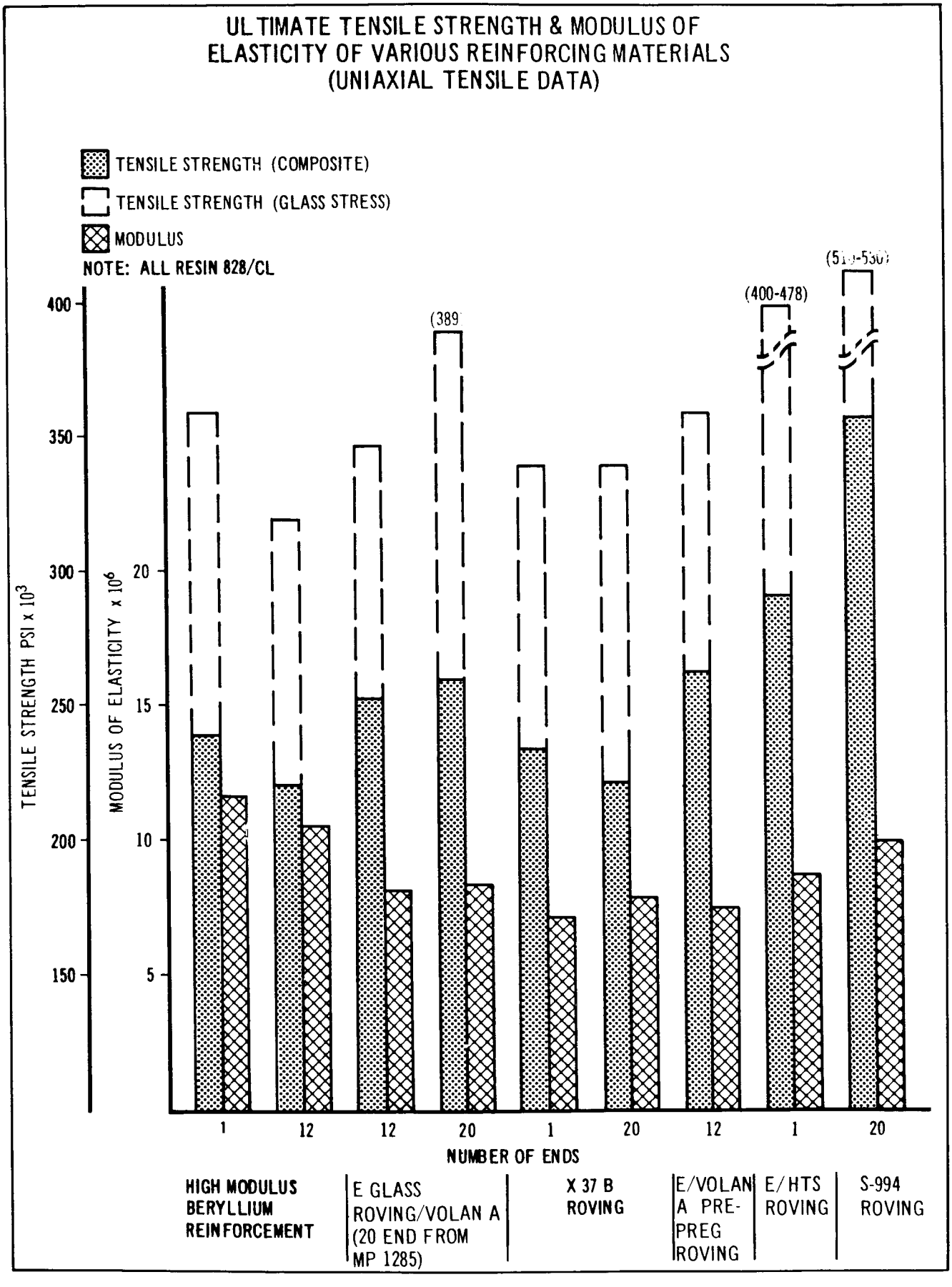

ULTIMATE TENSILE STRENGTH & MODULUS OF
ELASTICITY OF VARIOUS REINFORCING MATERIALS
(UNIAXIAL TENSILE DATA)
TENSILE STRENGTH (COMPOSITE)
TENSILE STRENGTH (GLASS STRESS)
MODULUS
NOTE: ALL RESIN 828/CL
TENSILE STRENGTH PSI x 10^3
MODULUS OF ELASTICITY x 10^6
400
350
300
250
200
150
20
15
10
5
(389
(400-478)
1
12
12
20
1
20
12
1
20
NUMBER OF ENDS
HIGH MODULUS BERYLLIUM REINFORCEMENT
E GLASS ROVING/VOLAN A (20 END FROM MP 1285)
X 37 B ROVING
E/VOLAN A PRE-PREG ROVING
E/HTS ROVING
S-994 ROVING

FIGURE A-3

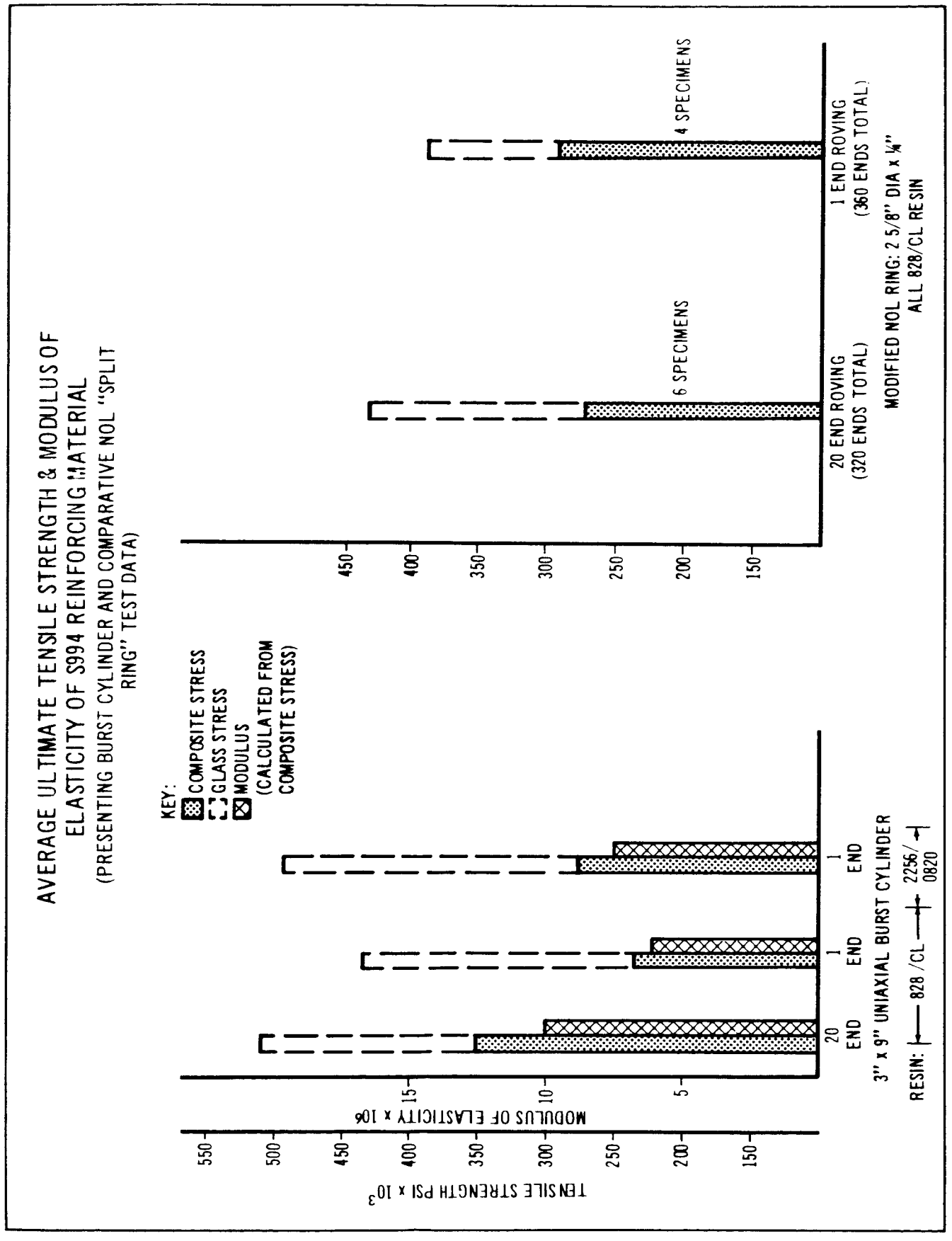

AVERAGE ULTIMATE TENSILE STRENGTH & MODULUS OF ELASTICITY OF S994 REINFORCING MATERIAL
(PRESENTING BURST CYLINDER AND COMPARATIVE NOL "SPLIT RING" TEST DATA)
KEY:
COMPOSITE STRESS
GLASS STRESS
MODULUS (CALCULATED FROM COMPOSITE STRESS)
TENSILE STRENGTH PSI x 10³
MODULUS OF ELASTICITY x 10⁶
20 END ROVING (320 ENDS TOTAL)
1 END ROVING (360 ENDS TOTAL)
6 SPECIMENS
4 SPECIMENS
MODIFIED NOL RING: 2 5/8" DIA x ¼"
ALL 828/CL RESIN
3" x 9" UNIAXIAL BURST CYLINDER
RESIN: 828 /CL 2256/0820

FIGURE A-4

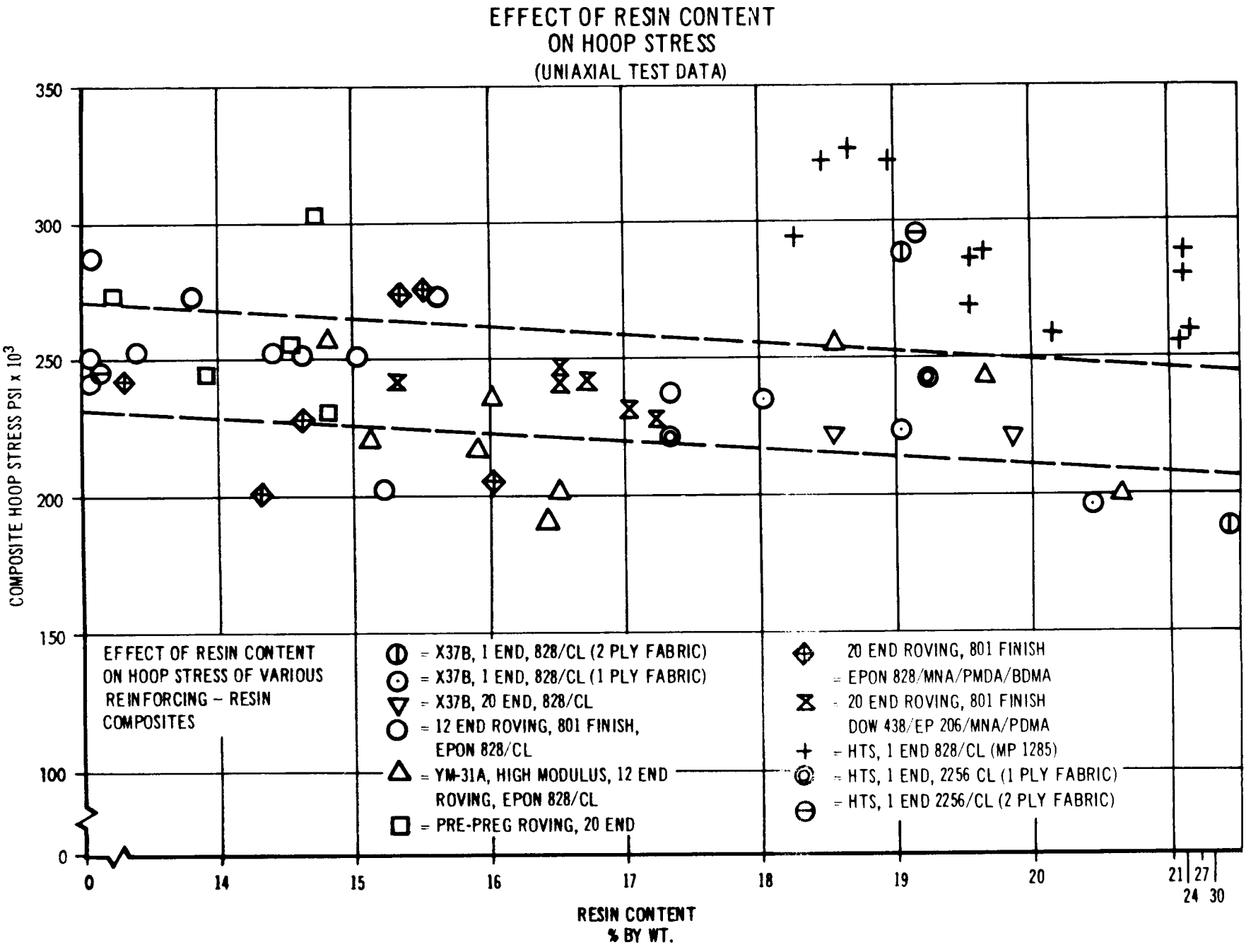

EFFECT OF RESIN CONTENT
ON HOOP STRESS
(UNIAXIAL TEST DATA)
COMPOSITE HOOP STRESS PSI x 10³
350
300
250
200
150
100
0
0
14
15
16
17
18
19
20
21
24
27
30
RESIN CONTENT
% BY WT.
EFFECT OF RESIN CONTENT ON HOOP STRESS OF VARIOUS REINFORCING – RESIN COMPOSITES
= X37B, 1 END, 828/CL (2 PLY FABRIC)
= X37B, 1 END, 828/CL (1 PLY FABRIC)
= X37B, 20 END, 828/CL
= 12 END ROVING, 801 FINISH, EPON 828/CL
= YM-31A, HIGH MODULUS, 12 END ROVING, EPON 828/CL
= PRE-PREG ROVING, 20 END
20 END ROVING, 801 FINISH = EPON 828/MNA/PMDA/BDMA
20 END ROVING, 801 FINISH DOW 438/EP 206/MNA/PDMA
= HTS, 1 END 828/CL (MP 1285)
= HTS, 1 END, 2256 CL (1 PLY FABRIC)
= HTS, 1 END 2256/CL (2 PLY FABRIC)

FIGURE A-5

APPENDIX B

EFFECTIVE GAGE LENGTH DETERMINATION FOR ORGANIC FILMS

APPENDIX B

EFFECTIVE GAGE LENGTH DETERMINATION FOR ORGANIC FILMS (UNIAXIAL TESTS)

The technique used for testing the dog-bone shaped specimens is as described by Smith (Ref. 30).

Two conditions are necessary for determining a relationship between crosshead travel of the testing machine and strain in the specimen. The crosshead displacement must be at a constant rate and the strain rate of the test section must also be constant. If these two conditions are met, it is possible to determine an "effective gage length", which is an equivalent straight rectangular length that would yield equivalent strains for the same crosshead travel.

Assuming that the crosshead rate is a constant and that the specimen strains at a constant rate in the test section, the following relationships can be determined.

let L = The distance between the jaws at any time (on a hypothetical straight specimen)

L_e = Effective gage length (the original distance between the jaws on the hypothetical specimen)

CS = Cross head speed

t = time

Then at any time t the cross head displacement is equal to:

$$L - L_e \text{ and } (CS)t$$

therefore $L - L_e = (CS)t$

and dividing by L_e

$$\frac{L - L_e}{L_e} = \frac{(CS)t}{L_e} \qquad (1)$$

Now let

D = Distance between any two bench marks on the dog-bone shaped specimen.

D_o = Original distance between the same two bench marks.

Then:
$$\frac{D - D_o}{D_o} = kt \qquad (2)$$

where k is the strain rate of the specimen.

Examination of Equations (1) and (2) shows that the following equation holds:

$$\frac{CS}{L_e} = k$$

or
$$L_e = \frac{CS}{k} \quad ;$$

rearrangement of equation (2)
yields

$$D = D_o\, k\, t + D_o \ ,$$

which is a straight line function.

Preliminary procedure was to draw bench marks on the specimens. In addition to the bench marks, axial and end lines which define the position of the specimen when installed in the grips were drawn on each specimen. The bench marks were drawn 1/32 inch thick to insure their sharp appearance in the time lapse photographs. The Instron Machine and photo equipment set-up are shown in Fig. B-1.

In order to meet the test method requirement of constant head rate, an Instron Model T.T. Universal Test Machine was used for the tests. The specimens were installed in the test fixture and the two upper pins removed. The arms were swung out of contact with the upper grips. As the tests were run, time lapse photographs of the test specimens were taken.

TEST SET-UP FOR ORGANIC FILMS

FIGURE B - 1

A Vanguard Motion Film Analyzer was utilized for measurement of separation of the bench marks with respect to time. The film viewer is equipped with movable vertical and horizontal cross hairs which are connected to cross hair travel indicators. The indicators enable reading distances on the film to the nearest .001 inch. A frame counter on the film analyzer was used to determine the time lapse from the start of the test to the frame being examined. From the photo analysis data, a distance between bench marks vs. time curve was plotted to establish the linearity of specimen strain rate, e.g. a specimen of Mylar "A" is shown in Fig. B-2. After linearity was established, the effective gage length was computed, e.g. Mylar "A" specimen, Fig. B-3. Subsequent reduction was accomplished numerically with a digital computer.

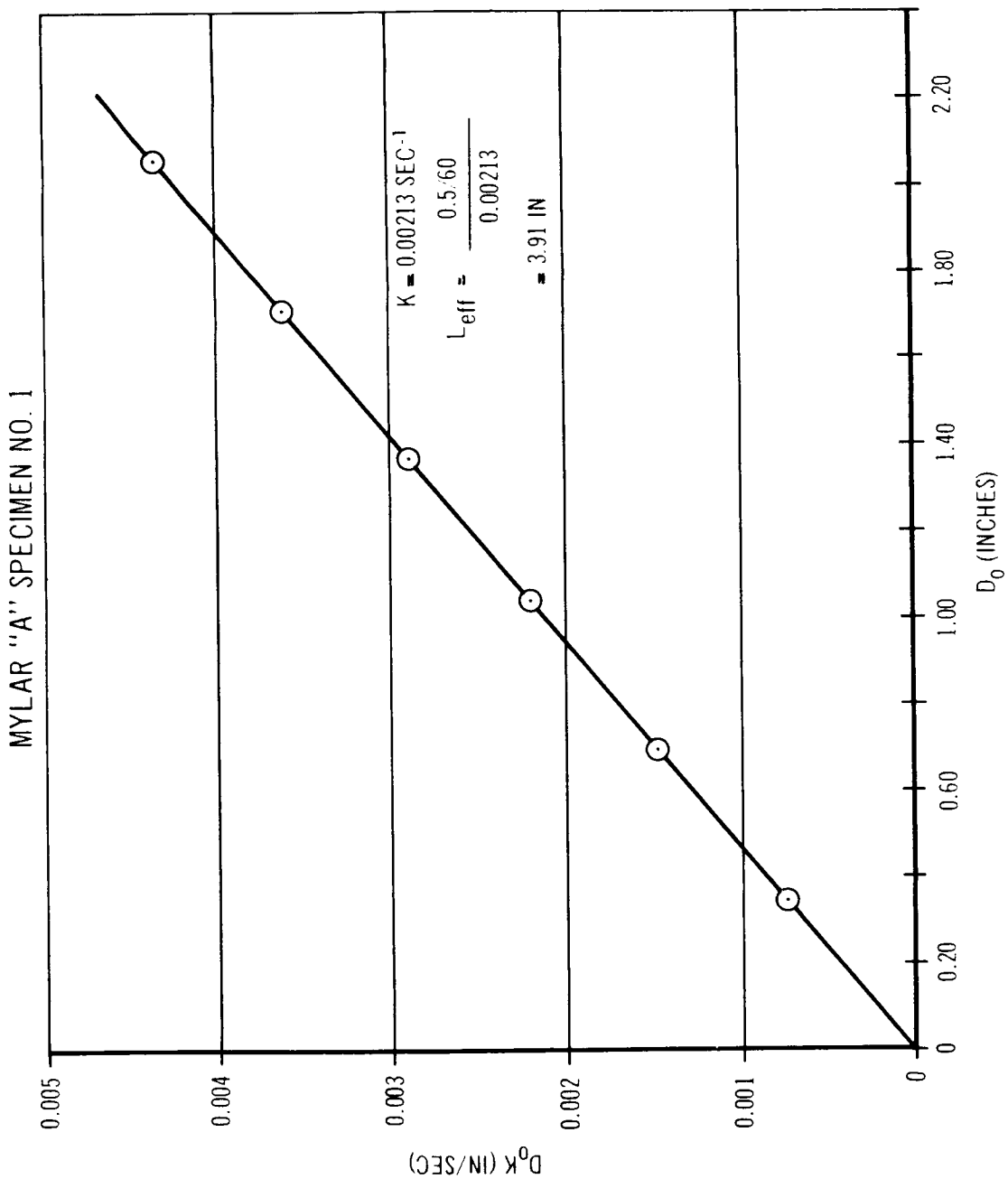

MYLAR "A" SPECIMEN NO. 1
K = 0.00213 SEC⁻¹
$L_{eff} = \frac{0.5/60}{0.00213}$
= 3.91 IN
D_0K (IN/SEC)
0.005
0.004
0.003
0.002
0.001
0
0
0.20
0.60
1.00
1.40
1.80
2.20
D_0 (INCHES)

FIGURE B-2

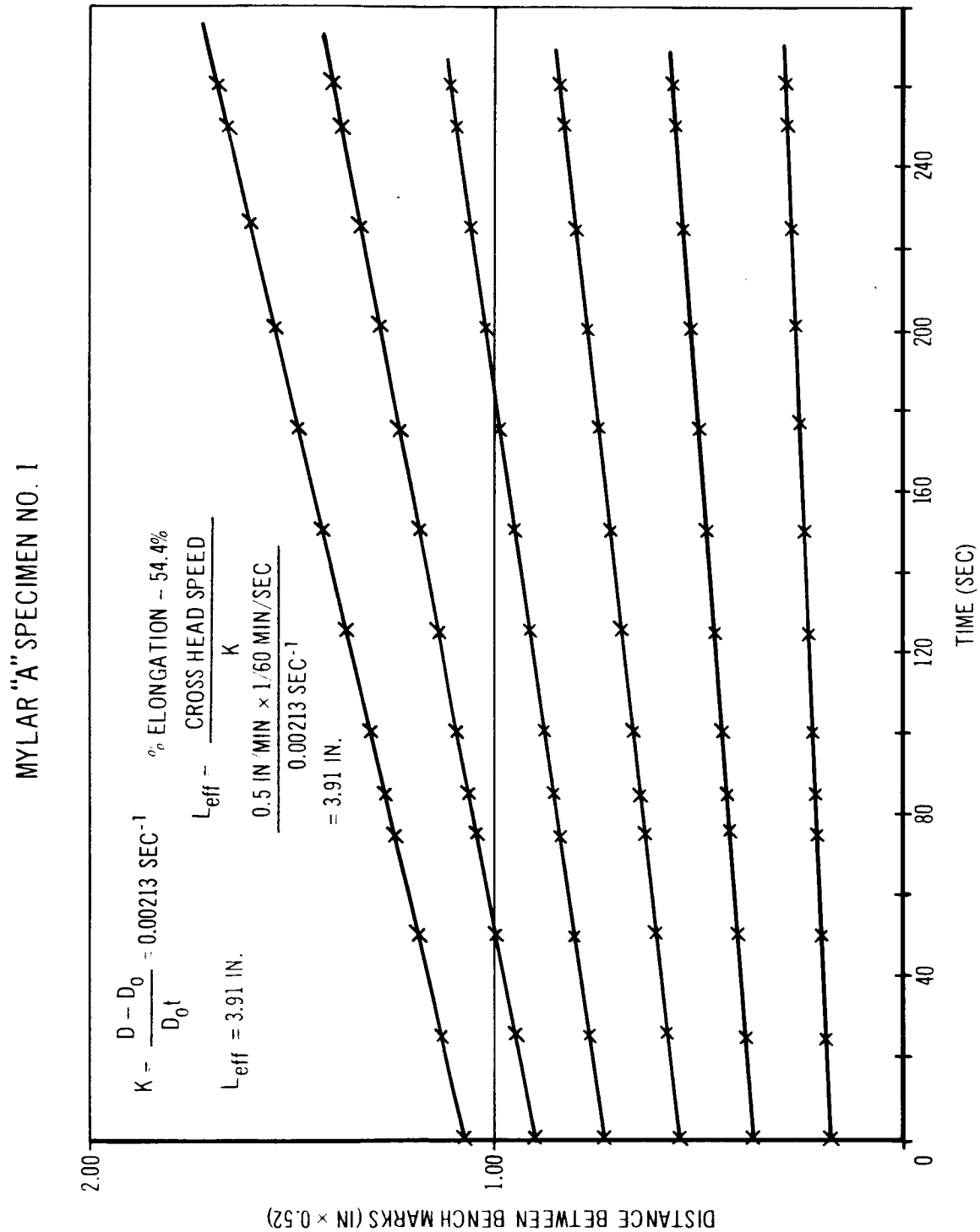

MYLAR "A" SPECIMEN NO. 1
$K = \frac{D - D_0}{D_0 t} = 0.00213\ SEC^{-1}$
% ELONGATION = 54.4%
$L_{eff} = 3.91$ IN.
$L_{eff} = \frac{\text{CROSS HEAD SPEED}}{K}$
$\frac{0.5\ IN/MIN \times 1/60\ MIN/SEC}{0.00213\ SEC^{-1}}$
= 3.91 IN.
2.00
1.00
0
40
80
120
160
200
240
TIME (SEC)
DISTANCE BETWEEN BENCH MARKS (IN × 0.52)

FIGURE B-3

APPENDIX C

UNIAXIAL MECHANICAL PROPERTIES

APPENDIX C

UNIAXIAL MECHANICAL PROPERTIES

		0.2% Yield psi	Ultimate psi	Modulus psi x 10^6	Ultimate Elongation %	Modulus 60% ult psi x 10^6
POLYURETHANE	R. T.	2140*	3560	**	249.92	0.00428
		2040*	3400	**	225.74	0.00408
		2210*	3680	**	244.74	0.00335
		2400*	4000	**	257.77	0.00267
		2040*	3360	**	202.36	0.00501
	-320 °F	16000	17700	0.667	3.16	0.665
		16500	18600	0.800	3.01	0.774
		16000	17400	0.653	3.38	0.633
		16400	18300	0.715	2.99	0.707
		15900	16700	0.662	2.94	0.645
	-423 °F	+	22000	0.956	2.43	0.923
		+	18400	0.943	1.99	0.941
		+	18100	1.240	1.41	1.240
		+	18000	0.797	2.26	0.800
		+	18100	0.617	2.11	0.910
GLASS FLAKES	R. T.	5172*	8620	**	39.50	1.150
		5760*	9600	**	72.28	1.231
		5292*	8820	**	53.68	1.058
		5592*	9320	**	61.07	1.118
		5460*	9100	**	54.03	1.215
	-320 °F	5750	7200	0.370	3.41	0.247
		6550	8030	0.500	3.14	0.321
		5700	7960	0.545	3.88	0.382
		5900	7460	0.500	3.54	0.332
		5800	7670	0.545	3.41	0.370
	-423 °F	3700	7980	0.750	2.07	0.501
		5600	9180	0.580	2.84	0.500
		4800	7900	0.800	2.42	0.593
		6100	8660	0.450	2.75	0.386
		4900	8250	0.667	2.81	0.495
"H" FILM	R. T.	12564*	20940	**	29.77	0.278
		10680*	17800	**	24.76	0.281
		10200*	17000	**	20.31	0.340
		9960*	16600	**	20.25	0.332
		10740*	17900	**	27.50	0.322
	-320 °F	+	27700	0.717	4.23	0.717
		+	22400	0.782	3.38	0.747
		+	23500	0.774	3.58	0.743
		+	27600	0.800	4.28	0.753
		+	18300	0.842	2.45	0.815
	-423 °F	+	16100	0.725	2.22	0.725
		+	16400	0.864	1.90	0.864
		+	21000	1.47	1.43	1.47
		+	13100	1.13	1.16	1.13
		+	15500	0.891	1.74	0.891

APPENDIX C

UNIAXIAL MECHANICAL PROPERTIES (CONT'D)

		0.2% Yield psi	Ultimate psi	Modulus psi x 10^6	Ultimate Elongation %	Modulus 60% ult psi x 10^6
TEDLAR BG 30 WH	R.T.	6060*	10100	**	111.20	0.0276
		6660*	11100	**	119.33	0.0167
		5940*	9900	**	100.17	0.0224
		5340*	8900	**	94.76	0.118
		5868*	9780	**	93.27	0.0266
	-320 °F	20500	31300	0.125	3.61	0.114
		20500	30400	0.125	3.45	0.114
		20000	29500	0.119	3.30	0.111
		21500	31400	0.122	3.67	0.111
		20500	29600	0.111	4.00	0.0986
	-423 °F	+	15100	0.157	0.965	0.157
		29500	32800	0.156	2.40	0.156
		+	26000	0.136	1.91	0.136
		29000	34500	0.147	2.93	0.147
		+	19800	0.141	1.40	0.141
MYLAR A	R.T.	13920*	23200	**	42.59	0.310
		11700*	19500	**	28.48	0.690
		13200*	22000	**	54.94	0.528
		15000*	25000	**	59.99	0.231
		14640*	24400	**	55.66	0.266
	-320 °F	27000	34700	0.833	4.94	0.817
		28500	36600	0.833	5.25	0.830
		27500	37200	0.910	5.21	0.859
		28000	38200	0.910	5.32	0.897
		28500	36200	0.910	4.82	0.873
	-423 °F	+	38400	1.250	0.44	1.095
		+	39000	0.714	0.75	0.650
		+	40500	0.625	0.78	0.594
			NO DATA			
		+	43200	0.555	0.94	0.552

* 60% of ultimate stress

** undefinable

\+ not applicable

APPENDIX C

UNIAXIAL MECHANICAL PROPERTIES (CONT'D)

		0.2% Yield psi	Ultimate psi	Modulus psi x 10^6	Ultimate elongation %
NICKEL	R.T.	61000	96300	43.2	7.45
		72500	103100	21.8	3.90
		75000	104200	19.9	2.30
		75000	105400	18.7	3.50
		85000	115500	23.2	3.98
	-320 °F	62000	98500	23.7	5.16
		86000	97500	21.4	4.08
		67000	101700	22.6	4.98
		74000	111000	25.0	5.94
		85000	119000	26.2	5.91
	-423 °F	53000	78800	19.3	2.70
		73000	91900	20.2	1.44
		76000	95000	19.1	1.48
		72000	92300	22.9	1.42
		83000	94100	19.5	1.44
COPPER	R.T.	19500	29600	7.9	6.45
		17000	30900	11.3	9.70
		17500	26400	10.1	4.50
		16000	31500	12.2	12.95
		15000	25400	8.9	7.90
	-320 °F	18000	30600	18.8	5.94
		19000	38400	13.1	5.76
		18500	29600	10.6	5.94
		19500	41100	7.5	6.24
		20500	29300	9.9	1.48
	-423 °F	20500	25400	16.1	1.41
		20500	27600	18.5	1.68
		29500	39000	33.2	2.27
		25500	29100	9.2	1.86
		20500	27300	14.9	2.13
SILVER	R.T.	24000	30400	11.2	12.76
		24750	33500	12.9	9.20
		22750	32200	8.3	17.75
		24250	29400	9.05	5.60
		25000	29550	7.7	7.70
	-320 °F	24500	35000	13.1	5.22
		32000	42300	9.8	5.16
		31000	42900	9.7	5.25
		29750	40000	11.3	7.14
		31500	43600	11.1	7.20
	-423 °F	22000	32500	10.0	4.38
		28500	39500	7.3	1.53
		25500	33000	7.7	1.54
		22000	31400	6.2	1.46
		24750	31900	8.3	1.41

APPENDIX D

LINER THERMAL CONTRACTION

APPENDIX D

LINEAR THERMAL CONTRACTION

MATERIAL	CONTRACTION 75°F to -103°F (10^{-3} in/in)	CONTRACTION 75°F to -320°F (10^{-3} in/in)	CONTRACTION 75°F to - 423°F (10^{-3} in/in)
Glass	1.12	2.33	2.39
Flake	1.10	2.20	2.30
Mylar "A"	1.85	3.56	3.89
	1.92	3.57	3.84
H-Film	2.56	4.96	5.45
	2.40	4.58	5.08
Tedlar	5.09	8.48	9.03
	5.34	8.50	9.29
Mylar "HS"	4.68	8.71	9.32
	4.69	8.76	9.69
Polyurethane	9.91	15.94	16.32
	9.64	15.65	16.31
FEP Teflon	11.03	17.16	---
	10.96	17.02	---
FEP Teflon (Ref.	10.50	15.80	17.00
Nickel	1.18	2.36	2.68
Plating	1.26	2.38	2.53
Nickel (Ref.	1.20	2.24	2.35
Copper	1.66	3.25	3.31
Plating	1.65	3.26	3.40
Copper (Ref.	1.58	3.10	3.33
Silver	1.87	3.88	4.44
Plating	1.87	3.85	4.32
Resin-	0.77	1.48	1.65
Fiber C	0.76	1.46	1.67
Resin-	0.76	1.62	1.84
Fiber B	0.85	1.68	1.82
Resin-	0.86	1.63	1.85
Fiber A	0.92	1.69	1.95

APPENDIX E

PROPERTIES OF PERTINENT GASES AND CRYOGENIC LIQUIDS

TABLE E - 1

VARIOUS PROPERTIES OF GASES

Gas	Atomic Weight (1959)	Molecular Diameter, 10^{-8} cm.			Average Velocity 100 cm/Sec		Collision Frequency 10^6
		From Viscosity	From Van Der Waal's Expansion	From Thermal Conductivity	0°C	20°C	20°C
Hydrogen	1.0080	2.40	2.34	2.32	1969	1775	10060
Helium	4.003	1.90	2.65	2.30	1208	1252	4540
Nitrogen	14.008	3.15	3.15	3.53	454	471	5070
Oxygen	16.000	2.98	2.92	----	425	440	4430

References:

Handbook of Chemistry and Physics
44th Edition
Chemical Rubber Publishing Company
Cleveland, Ohio - 1962

TABLE E - 2

PROPERTIES OF SEVERAL CRYOGENIC LIQUIDS

PROPERTY	HELIUM (He)	HYDROGEN (H_2)	NITROGEN (N_2)	OXYGEN (O_2)
Boiling point (1 ATM)	-452.0° F	-422.9° F	-320.4° F	-297.4° F
Melting point (1 ATM)		-434.4° F	-345.8° F	-361.0° F
Color	Colorless	Colorless	Colorless	Colorless
Odor	Odorless	Odorless	Odorless	Odorless
Liquid Density at B.P.	7.80 lb/ft^3	4.43 lb/ft^3	50.4 lb/ft^3	72.1 lb/ft^3
Vapor Density at B.P.	1.06 lb/ft^3	0.08 lb/ft^3	0.28 lb/ft^3	0.30 lb/ft^3
Heat of Vaporization at B.P.	69 BTU/ft^3	854 BTU/ft^3	4319 BTU/ft^3	6522 BTU/ft^3
Critical Density	4.32 lb/ft^3	1.89 lb/ft^3	19.4 lb/ft^3	26.9 lb/ft^3
Critical Temperature	-450.4°F	-400.2° F	-232.8° F	-181.8° F
Critical Pressure	33.2 psia	188 psia	492 psia	731 psia
Major Safety Hazards	Low temperature burns. Excess pressure due to boil-off	Fire and/or explosion when exposed to air, oxygen, or other oxidizers, low temperature burns	Low temperature burns. (Frostbite)	Fire and/or explosion with organic materials & with many inorganic materials, under severe impact, low temperature burns.

APPENDIX F

MATERIALS

APPENDIX F

MATERIALS

MYLAR

Mylar is a biaxially oriented polyester film. The film is tough, durable, and relatively impermeable to a number of organic and inorganic gases. Type "HS" is a heat shrinkable film, often used for packaging applications. The "HS" type exhibits a low per cent crystallinity of 15% compared to 30% crystallinity level for Mylar "A". Type "A" is a film with a low fault count (a measure of pinholes) for general use where a strong, durable material is required. This material proved to be the best plastic among those tested in the Douglas preliminary program (approximately 20). The excellent strength, elongation, and cyclic strain factors evident in preliminary testing were verified by recent testing results.

(Manufacturer: E. I. duPont de Nemours & Co., Film Dept.
4455 Fruitland Avenue
Los Angeles 58, California)

TEDLAR BG30WH

Tedlar, a polyvinyl fluoride film, has similar properties to Mylar but with the added advantage of being able to be formed. Permeability to hydrogen at room temperature reports to be less than half that of Mylar. Physical properties though not as good as Mylar appear sufficient. "BG30WH" indicates that both surfaces of the film are bondable, the surface finish is glossy, the strength medium, and the color white. The per cent crystallinity level of Tedlar is 30 - 40%.

(Manufacturer: E. I. duPont de Nemours & Co., Film Dept.
4455 Fruitland Avenue
Los Angeles 58, California)

H-FILM

H-Film, a new DuPont developed polyimide film, appears as promising for low temperature as well as high temperature use for which it was developed. The mechanical properties appear comparable to Mylar at room temperature. There are reports of extremely low temperature flexibility behavior and any material exhibiting good properties from extremely high temperatures to extremely low temperatures is worth examining. H-Film is reported to have a per cent crystallinity level of from 0 to 3%. However, H-Film is known to vary in crystallinity even in the same roll.

(Manufacturer: E. I. duPont de Nemours & Co., Film Dept.
Yerkes Research & Development Laboratory
Buffalo 7, New York)

SEILON UR29E

UR29E, is a polyurethane film exhibiting the tough, notch resistance, abrasion resistance, high elongation, and chemical resistance, expected of polyurethanes. This material can easily be formed and sealed and from a processing stand-point, seems ideal as a liner. However, its high permeability rate is a detrement. Information is currently unavailable concerning per cent crystallinity of Seilon UR29E.

(Manufacturer: Seiberling Rubber Co.
Distributor: Lustro Corp. of Calif.
810 East Third St.
Los Angeles 13, California)

GLASS FLAKES

Glass Flakes of random size and 5 micron thickness, "E" composition (Silicon Dioxide, Calcium Oxide, Aluminum Oxide, Boron Oxide, and maybe Sodium and Potassium Oxide, and Magnesium Oxide), used in a composite with either epoxy, resin, polyurethane resin, celluose carrier, or polyester-celluose carrier promises to be an excellent barrier film. The flat plate structure of the glass flakes increases the path which the gas or cryogen must travel to

penetrate a given film thickness.

(Manufacturer: Owens-Corning Fiberglas, Aerospace Division
5933 Telegraph Road
Los Angeles 22, California)

ELECTROFORMED NICKEL

This material is easily deposited and has the required material properties at room temperature. The coefficient of expansion of nickel is very near that of a fiber glass/laminate and the elongation at -423°F is over 20%. Strength is high.

(Manufacturer: Electroforms, Inc.
239 E. Gardena Blvd.
Gardena, California)

ELECTROFORMED COPPER

This material is also easily deposited and has the required material properties at room temperature. It has lower strength than the nickel but slightly higher elongation. The coefficient of thermal contraction is slightly higher than that of nickel.

(Manufacturer: Electroforms, Inc.
239 E. Gardena Blvd.
Gardena, California)

ELECTROFORMED SILVER

This material is also easily deposited. The room temperature strength (15,000 psi) is rather low compared to the other two selected metals. Elongation at room temperature of 23% and the elongation of 38% in liquid oxygen are its best characteristics.

(Manufacturer: Electroforms, Inc.
239 E. Gardena Blvd.
Gardena, California)

PURE LEAD FOIL

Lead Foil Sheeting is used in combination with a plastic film because of the ease of forming lead foil over a double contoured surface. Lead, as most metallics, is impermeable to gases and bonds readily to fiber glass.

(Distributor: Crawford Foil Co.
5920 Blackwelder St.
Culver City, California)

S994/HTS SCG150 1/0 1.0 Z FIBER GLASS

S994/HTS SCG150 1/0 1.0 S FIBER GLASS

S994/HTS Fiber Glass is used since it represents the most advanced product available and its mechanical properties are superior to E glass, HTS finish.

S & Z twist are used to provide balanced construction.

(Manufacturer: Owens-Corning Fiberglas, Aerospace Division
5933 Telegraph Road,
Los Angeles 22, California)

S994/HTS 12 END ROVING

S994/HTS 12 End Roving is used when time and size negates the use of single end yarn.

(Manufacturer: Owens-Corning Fiberglas, Aerospace Division
5933 Telegraph Road
Los Angeles 22, California)

FIBERGLASS CLOTH

Style #181 is used as a standard laminating cloth.

Style #1584 is used when a thicker laminate per layer is required.

Style #120 is used when a high density of glass per layer is required.

These cloths are standard "E" glass, Volan A finish.

Style #9943, S994/HTS with a 1543 construction, is used where unidirectional strength of the laminate (such as the longitudinal reinforcement) is required. The S994/HTS material provides better balanced strength with S994/HTS Fiberglas used in circumferential winding.

Style #181
Style #120
Style #1584
- Coast Manufacturing & Supply
Fabrics Division
4600 Shelia St.
Los Angeles 22, California

Style #9943 - Clark-Schwebel Fiberglass Corp.
2838 East Pico Blvd.
Los Angeles 23, California

NARMCO 7343/7139

Narmco 7343 is a polyurethane adhesive cured with Narmco 7139 (MOCA). This adhesive system is a revolutionary two-part system designed for cryogenic applications. Bonding of organic films, even hard-to-bond films such as Mylar, and curing can be accomplished under ambient conditions at contact pressure. Narmco 7343 is designed to produce tough mechanical joints even when exposed to cryogens. As an adhesive, the viscosity of the system is approximately that of black-strap molasses. To use as a winding resin a solvent or thinner such as MEK can be added. However, the disadvantages of not having a 100% solid resin system are present.

(Manufacturer: Narmco Materials Division
600 Victoria Street
Costa Mesa, California)

EC 2216 B/A

3M produces this modified (filled) epoxy adhesive. This adhesive exhibits good low temperature properties and as a result of experimental work, appears to bond H-Film better than the Narmco adhesive at cryogenic temperatures.

(Manufacturer: Minnesota Mining & Manufacturing
6411 Randolph Street
Los Angeles 22, California)

EPI-REZ 510

Epi-Rez 510 is a commercial grade of diglycidyl ether of bisphenol-A. The resin contains no modifier or diluent. Epi-Rez 510 has a very low hydrolyzable chlorine content. Epi-Rez 510 is the basic resin used as the starting point for most epoxy resin applications. The chlorine content is approximately o.3%. The weight per epoxide is 185 - 200.

(Manufacturer: Jones-Dabney Co.
Resins & Chemicals Division
3951 Medford St.
Los Angeles 63, California)

EPI-REZ 5101

Epi-Rez 5101 is a highly purified low chlorine content version of the standard resin, Epi-Rez 510. Jones-Dabney manufacturing specification calls for a minimum of 0.1% total chlorine content. The weight per epoxide is 185 - 200.

(Manufacturer: Jones-Dabney Co.
Resins & Chemicals Division
3951 Medford Street
Los Angeles 63, California)

EPI-REZ 5042

Epi-Rez 5042 is an epoxy resin based on an aliphatic polyol. The use of Epi-Rez 5042 in conjunction of other resins such as Epi-Rez 510 are as follows:

1) Epi-Rez 5042 will accelerate the curing rate
2) Epi-Rez 5042 will lower the viscosity of the system
3) The wetting and leveling properties of the system are improved

4) Epi-Rez 5042 imparts better flexibility and impact characteristics. The epoxy equivalent weight is 138 - 153.

(Manufacturer: Jones-Dabney Co.
Resins & Chemicals Division
3951 Medford Street
Los Angeles 63, California)

EPI-REZ 5085

Epi-Rez 5085 is a medium viscosity 100% reactive epoxy resin based on the reaction of epichlorohydrin and bisphenol-A.

It is internally modified to improve the flexibility of the cured product. Another characteristic of this resin is the longer pot life obtainable than with the unmodified Epi-Rez 510 type resin. The epoxy equivalent weight is 355-385.

(Manufacturer: Jones-Dabney Co.
Resins & Chemicals Division
3951 Medford Street
Los Angeles 63, California)

EPI-REZ 502

Epi-Rez 502 is a new low viscosity diepoxide resin which is aliphatic in nature. This material is sometimes used to replace a portion of Epi-Rez 510 to increase flexibility and reduce viscosity. In room temperature curing systems, it will not alter the curing rate and will improve adhesion. The weight per epoxide is 300-335.

(Manufacturer: Jones-Dabney Co.
Resins & Chemicals Division
3951 Medford Street
Los Angeles 63, California)

EPI-CURE 841

Epi-Cure 841 is a liquid mixture of aromatic polyamines containing methylene dianiline which provide the same properties and chemical resistance obtainable with aromatic amine curing agents without the inconvenience of handling a solid curing agent. The reaction of Epi-Cure 841 with epoxy resins at room temperature is quite slow, and since no heat is required to dissolve the curing agent in the resin, the pot life is considerably longer than that of equivalent mixtures catalyzed with a solid aromatic amine. This hardener is B-stagnable.

(Manufacturer: Jones-Dabney Co.
Resins & Chemicals Division
3951 Medford St.
Los Angeles 63, California)

EPI-CURE 855

Epi-Cure 855 is an aliphatic amido-amine, moderately reactive curing agent which provides room temperature curing systems having extended pot life, lower viscosity, and complete compatibility with conventional epoxy resins. The low viscosity of Epi-Cure 855 eliminates the need for reactive diluents in the resin system.

(Manufacturer: Jones-Dabney Co.
Resins & Chemicals Division
3951 Medford St.
Los Angeles 63, California)

ERLA 0510

ERLA 0510 is essentially the monomeric triglycidyl derivative of para-amino phenol. This product is a highly distilled version of ERLA 0500. The molecular distillation removes some of the hydroxyl groups present in ERLA 0500, thereby extending the pot life. ERLA 0510 also reports an increased heat distortion point through its compact molecular structure and pure trifunctionality. The epoxy equivalent weight is 185-200.

(Manufacturer: Union Carbide Plastics Co.
2770 Leonis Blvd.
Los Angeles 58, California)

ERL 2772

ERL 2772 is a low viscosity version of Bakelite's standard ERL 2774 bisphenol-A type resin. An epoxy equivalent weight of 185 - 195 is reported. A low per cent of hydrolyzable chlorine, 0.18% is also reported. This resin is a general purpose, low viscosity epoxy resin.

(Manufacturer: Union Carbide Plastics Co.
2770 Leonis Blvd.
Los Angeles 58, California)

ERL 2256

ERL 2256 is a difunctional, low viscosity resin, with excellent handling properties. ERL 2256, when cured with appropriate hardeners, exhibits significant physical property improvements over the standard diglycidyl ether of bisphenol-A resins. This resin has excellent pot life characteristics. The epoxy equivalent weight is 130-150.

(Manufacturer: Union Carbide Plastics Co.
2770 Leonis Blvd.
Los Angeles 58, California)

ZZL 0822

ZZL 0822 is a flexible curing agent recommended for high impact strength applications. This hardener contains primary amines and a long, flexible molecular structure. This hardener is water soluble. Generally this hardener is used for flexibility.

(Manufacturer: Union Carbide Plastics Co.
2770 Leonis Blvd.
Los Angeles 58, California)

ZZL 0820

ZZL 0820 is a modified eutectic blend of aromatic amines. This hardener requires an elevated temperature cure and imparts good physical properties for ERL 2256.

(Manufacturer: Union Carbide Plastics Co.
2770 Leonis Blvd.
Los Angeles 58, California)

ZZL 0803

ZZL 0803 is a modified diethylene triamine (DETA) hardener whose functionality has been reduced from 5 to 3 with a polar nitrile group introduced by condensing one mole of DETA with two moles of acrylonitrile. The resultant product, ZZL 0803 is a low viscosity liquid capable of curing at room temperature with epoxy resins imparting good physical properties to the resin.

(Manufacturer: Union Carbide Plastics Co.
2770 Leonis Blvd.
Los Angeles 58, California)

EPON ERX 36

Epon Resin ERX 36 is a new epoxy resin developed especially for use in laminating, filament winding, and casting applications. ERX 36 has a lower viscosity, and longer pot life than a standard bisphenol-A type resin such as Epon 828. An important practical advantage for an Epon ERX 36 / CL system is that precise control over curing agent stoichiometry is not critical. The weight per epoxide equivalent is 192.

(Manufacturer: Shell Chemical Co.
Plastics & Resins Division
10642 Downey Ave.
Downey, California)

CL

CL (m-phenylenediamine) curing agent is a polyfunctional aromatic amine. This hardener has been used extensively with standard bisphenol-A resin.

(Distributor: Shell Chemical Co.
Plastics & Resins Division
10642 Downey Ave.
Downey, California)

DER 332

This resin is a high purity diglycidyl ether of bisphenol-A having a lack of polymer fractions. DER 332 assures, due to the purity, uniform performance and exceptionally low viscosity. The uniqueness of DER 332 epoxy resin is reflected in its maximum epoxide equivalent weight of 178 (172 - 178). Chemically pure diglycidyl ether of bisphenol-A would report 170.

(Manufacturer: Dow Chemical Co.
305 Crenshaw Blvd.
Torrance, California)

APCO 322

Apco 322 is a high heat distortion, aromatic, epoxy resin hardener to produce excellent toughness, long pot life, and low temperature cure. This hardener is a complex, highly functional polyamine of an aromatic nature with no aliphatic side chains or aliphatic diamines present.

(Manufacturer: Applied Plastics Company
130 Penn St.
El Segundo, California)

MASTER SUMMARY SCHEDULE FOR INVESTIGATION OF STRUCTURAL PROPERTIES OF FIBER GLASS FILAMENT-WOUND PRESSURE VESSELS AT CRYOGENIC TEMPERATURES

(Revised 9-30-63)

Prepared & Submitted By:

J. M. Toth, Jr.

J. M. Toth, Jr.
Investigation Director
AST/Structures Branch

Prepared For:
NASA/Lewis Research Center
Cleveland, Ohio

Contract NAS 3-2562
Control Number APO 2014

24 October 1963
A-260-D560-274

Approved By:

C. Y. Kam

C. Y. Kam, Chief
Structural Development Section
Advance Space Technology

Approved By:

H. H. Dixon

H. H. Dixon, Chief
Structures Branch
Advance Space Technology

MASTER SUMMARY SCHEDULE

(Revised 9-30-63)

1. A detailed description of each task area and method of investigation is delineated in the proposal, Douglas Santa Monica Report SM-43264; results to date and work to be accomplished during the next quarter are given in Douglas Santa Monica Report SM-45762.

   The revised schedule of tasks is given in Figures 1 through 4. The following milestones are to be noted:

	Original (7-29-63)	Revised
Authority to Proceed	July 1, 1963	July 1, 1963
Coupon testing completed	Sept. 6	Sept. 19
Subscale pressure vessel testing. Completed	Oct. 23	Dec. 27
First 18 inch diameter by 24 inch long pressure vessel completed	Nov. 4	Jan. 8, 1964
First 18 inch diameter by 24 inch long pressure vessel tested in LH_2	Nov. 12	Jan. 15
Phase I completed	Nov. 28	Feb. 4
First pressure vessel completed in phase II	Dec. 23	Feb. 11
First Phase IV test	Dec. 30	Feb. 18
First Phase III test	Feb. 12, 1964	Mar. 17
Phase IV completed	Sept. 23	Sept. 29
Phase II completed	Oct. 14	Oct. 20
Phase III completed	Oct. 21	Oct. 27
Technical Program completed	Oct. 31	Oct. 31
Program completed	Nov. 30	Nov. 30

2. Total manhour estimate and actual expenditure to date to perform the given Tasks are shown in Figure 5. The increased total manhours (17, 174 vs 16, 331) reflect fabrication of the subscale pressure vessels by Manufacturing rather than Engineering Laboratory personnel; also conversion of $4000 of the cryogenic reservoir test system helium into manhours due to the new cryogenic pump test system.

3. Manhour estimates and actual expenditures to date to perform the given Tasks are shown in Figures 6 through 12.

4. The Projected Funding Schedule and actual expenditure to date are shown in Figure 13.

NAS 3-2562
TOTH

TASK SCHEDULE

PHASE I

REV. A
9/30/63

JULY 5 10 15 20 25 | AUGUST 5 10 15 20 25 | SEPTEMBER 5 10 15 20 25 | OCTOBER 5 10 15 20 25 | NOVEMBER 5 10 15 20 25 | DECEMBER 5 10 15 20 25

COUPONS

UNIAXIAL TENSILE TESTING (LINERS) *

COEFFICIENT OF CONTRACTION AND DENSITY TESTING (LINERS AND RESIN/FIBER GLASS COMPOSITES) *

UNSTRESSED SPECIMEN ROOM TEMPERATURE GASEOUS NITROGEN AND HYDROGEN PERMEABILITY (LINERS) *

UNIAXIAL CYCLIC TENSILE - LIQUID HYDROGEN (RESIN/FIBER GLASS COMPOSITES)

FABRICATION *

TESTING *

7½ INCH DIAMETER BIAXIAL SUBSCALE PRESSURE VESSELS

ROOM TEMPERATURE

FABRICATION *

TESTING

LIQUID HYDROGEN

FABRICATION *

TESTING

LIQUID NITROGEN

FABRICATION

TESTING

18 INCH DIAMETER BY 24 INCH LONG PRESSURE VESSELS

FABRICATION

TESTING

JULY 5 10 15 20 25 | AUGUST 5 10 15 20 25 | SEPTEMBER 5 10 15 20 25 | OCTOBER 5 10 15 20 25 | NOVEMBER 5 10 15 20 25 | DECEMBER 5 10 15 20 25

1963

CODE

* WORK COMPLETE AS OF REVISION DATE

DESIGN, TOOLING, HOOK-UP, CHECK-OUT, ETC.

COMPLETED TASK (FAB OR TEST)

FIGURE 1

NAS 3-2562
TOTH

TASK SCHEDULE

PHASE I (CONT'D)

REV. A
7/30/63

JANUARY 5 10 15 20 25 | FEBRUARY 5 10 15 20 25 | MARCH 5 10 15 20 25 | APRIL 5 10 15 20 25 | MAY 5 10 15 20 25 | JUNE 5 10 15 20 25

18 INCH DIAMETER BY 24 INCH LONG PRESSURE VESSELS
LIQUID HYDROGEN

FABRICATION

TESTING

JANUARY 5 10 15 20 25 | FEBRUARY 5 10 15 20 25 | MARCH 5 10 15 20 25 | APRIL 5 10 15 20 25 | MAY 5 10 15 20 25 | JUNE 5 10 15 20 25

1964

FIGURE 2

NAS 3-2562
TcTH

TASK SCHEDULE

Rev. A
9/30/63

JANUARY 5 10 15 20 25 | FEBRUARY 5 10 15 20 25 | MARCH 5 10 15 20 25 | APRIL 5 10 15 20 25 | MAY 5 10 15 20 25 | JUNE 5 10 15 20 25

PHASE II
FAB VESSELS

VESSEL 3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22

PHASE IV

VESSEL 3
4
7
TEST AT -423°F 8
11
12
15
16
19
20

PHASE III

VESSEL 5
6
9
TEST AT -320°F 10
13
14
17
18
21
22

JANUARY 5 10 15 20 25 | FEBRUARY 5 10 15 20 25 | MARCH 5 10 15 20 25 | APRIL 5 10 15 20 25 | MAY 5 10 15 20 25 | JUNE 5 10 15 20 25

1964

6 MONTHS BY DAYS
X 90 DIVNS. JAN.-JUNE
KEUFFEL & ESSER CO.
359-137L

FIGURE 3

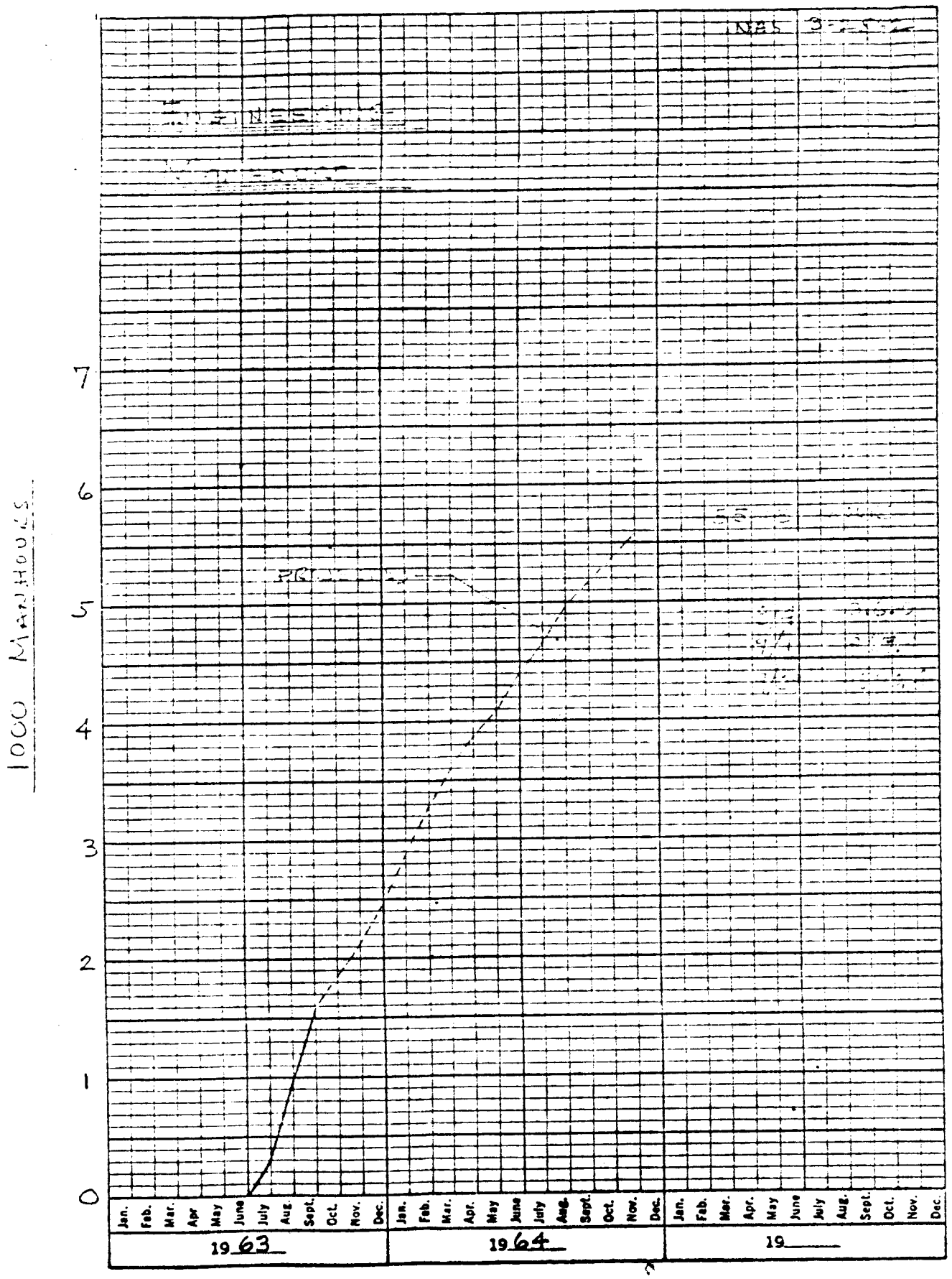

1000 MANHOURS
0
1
2
3
4
5
6
7
Jan. Feb. Mar. Apr May June July Aug Sept. Oct. Nov. Dec.
1963
Jan. Feb. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec.
1964
Jan. Feb. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec.
19

FIGURE 6

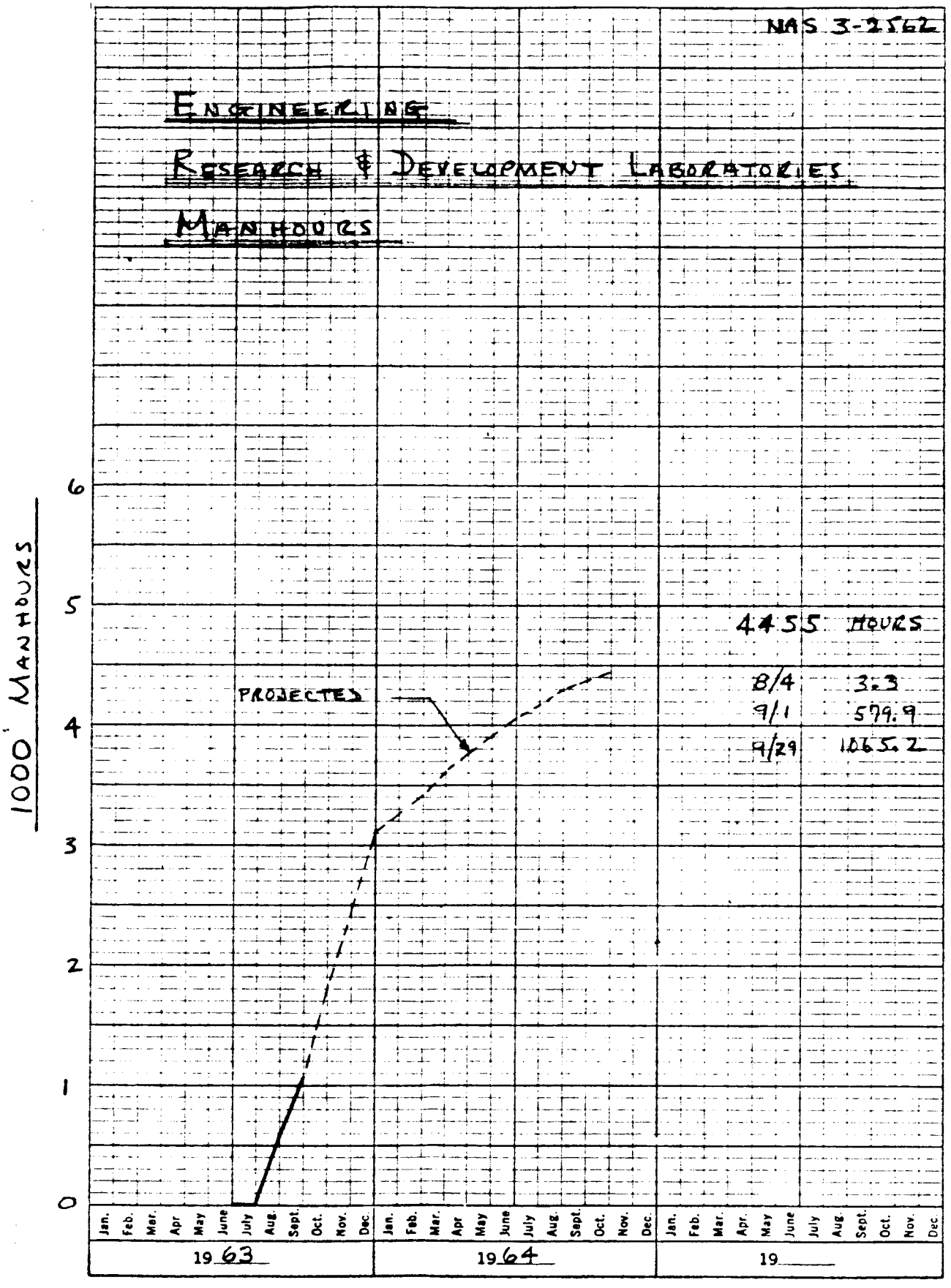

NAS 3-2562
ENGINEERING
RESEARCH & DEVELOPMENT LABORATORIES
MANHOURS
1000 MANHOURS
6
5
4
3
2
1
0
4455 HOURS
8/4 3.3
9/1 579.9
9/29 1065.2
PROJECTED
Jan. Feb. Mar. Apr May June July Aug. Sept. Oct. Nov. Dec.
1963
1964
19

FIGURE 7

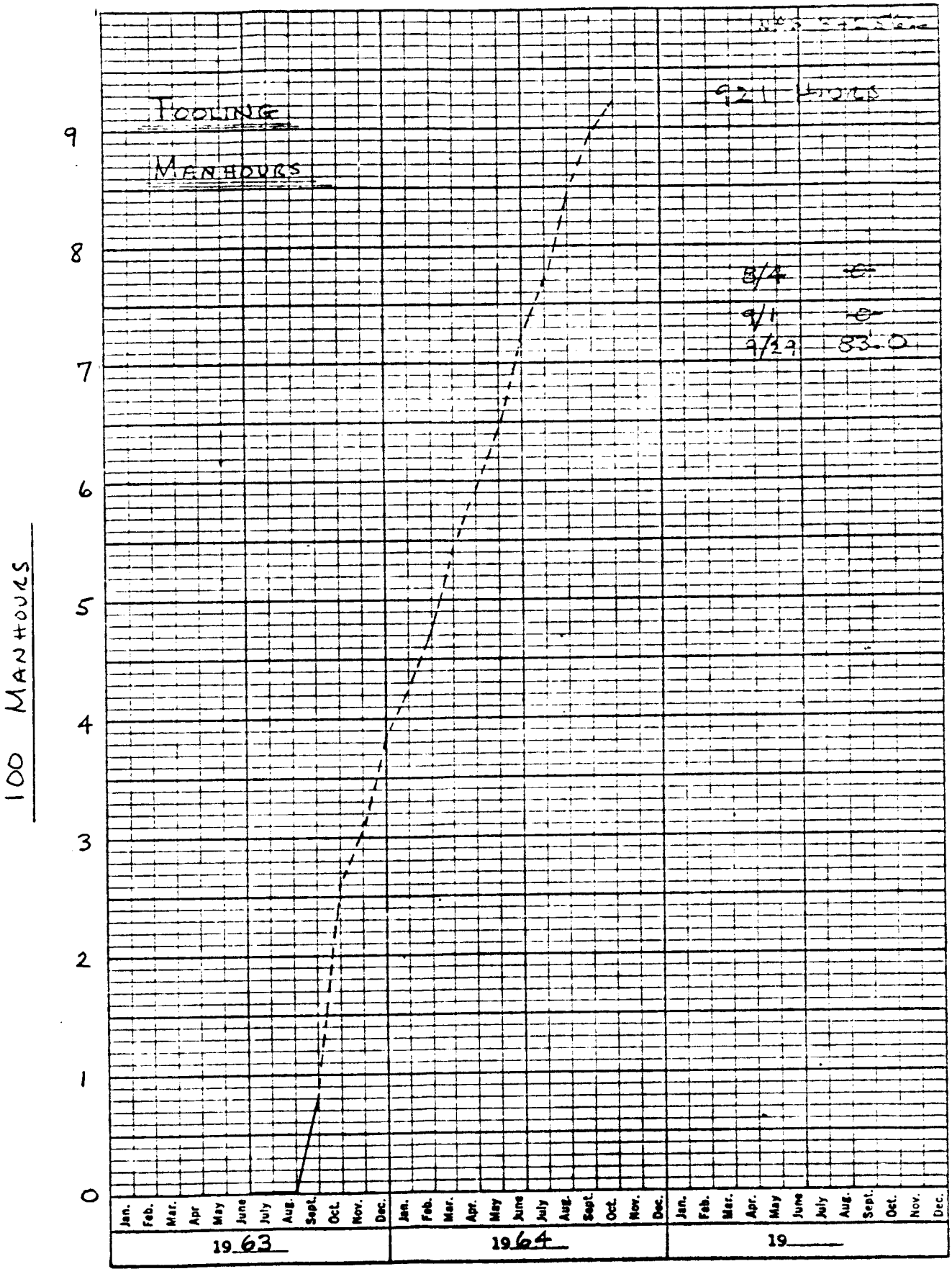

TOOLING
MANHOURS
100 MANHOURS
9
8
7
6
5
4
3
2
1
0
8/4
9/1
9/12
83.0
Jan. Feb. Mar. Apr May June July Aug. Sept. Oct. Nov. Dec.
1963
Jan. Feb. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec.
1964
Jan. Feb. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec.
19

FIGURE 8

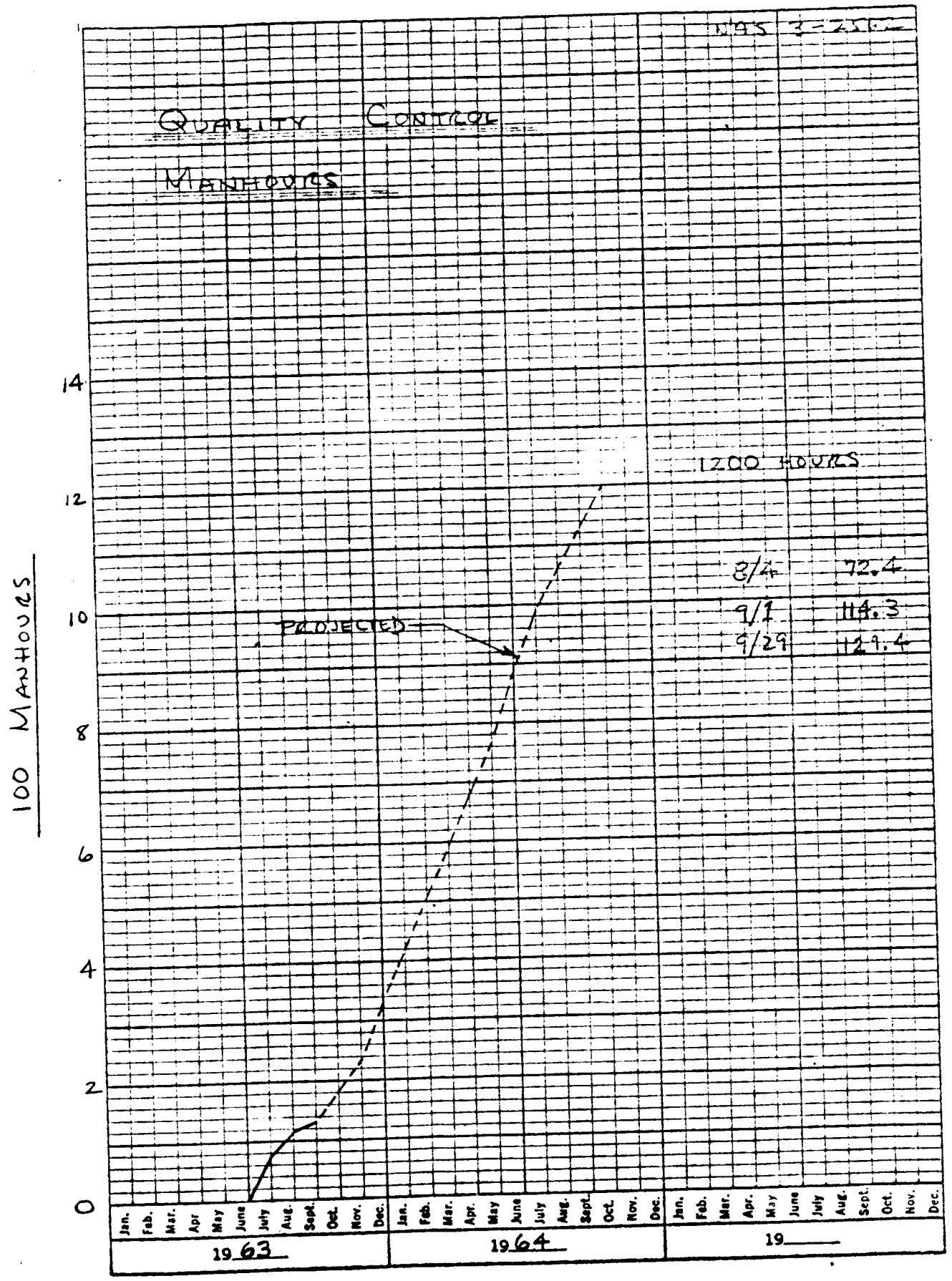

QUALITY CONTROL
MANHOURS
1295 3 = 255
1200 HOURS
8/4 72.4
9/1 114.3
9/29 129.4
PROJECTED
100 MANHOURS
0
2
4
6
8
10
12
14
Jan. Feb. Mar. Apr May June July Aug. Sept. Oct. Nov. Dec.
1963
1964
19

FIGURE 9

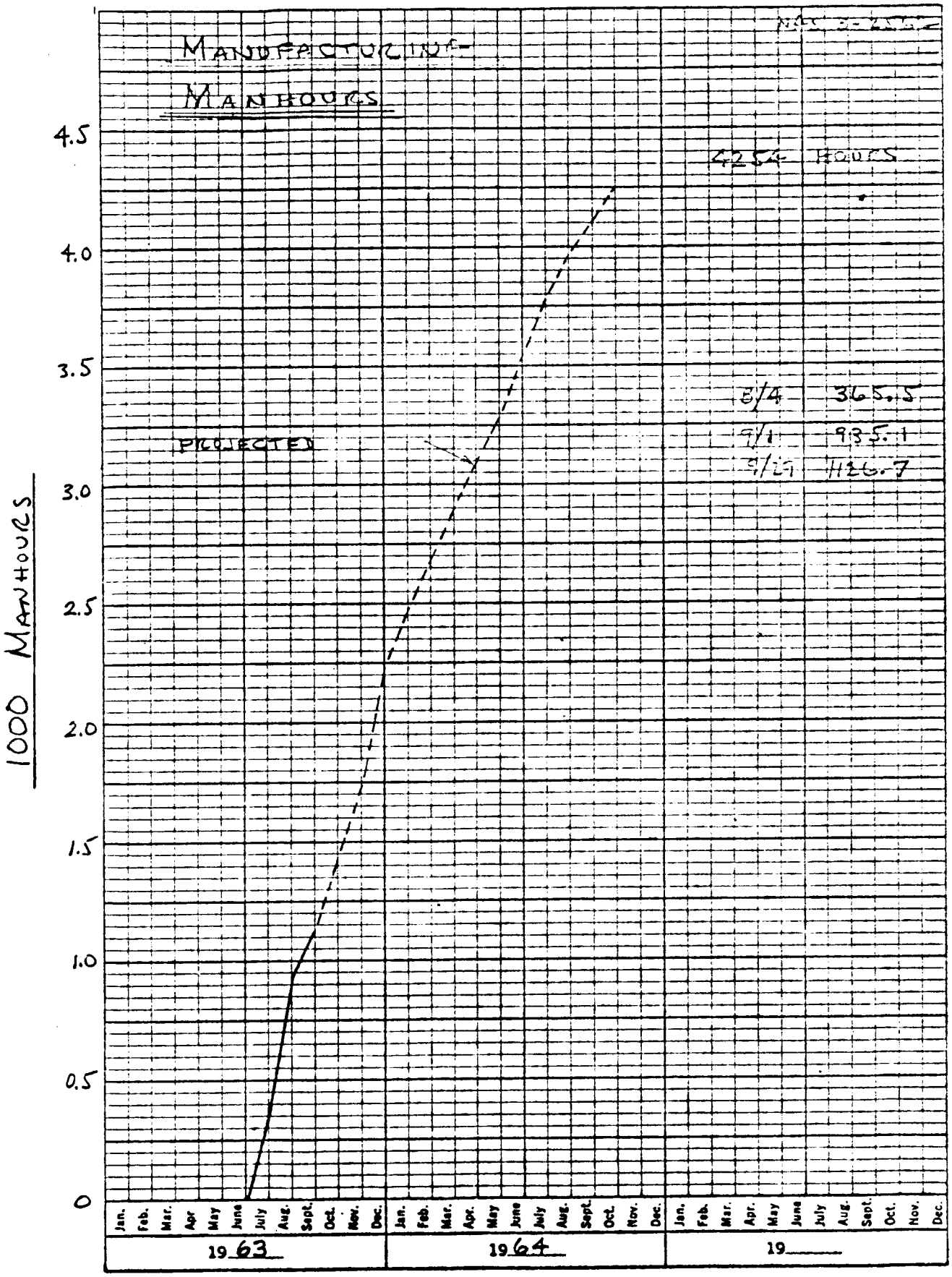

MANUFACTURING MANHOURS
4254 HOURS
8/4 365.5
9/1 935.1
9/29 426.7
PROJECTED
1000 MANHOURS
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Jan. Feb. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec.
1963
1964
19

FIGURE 10

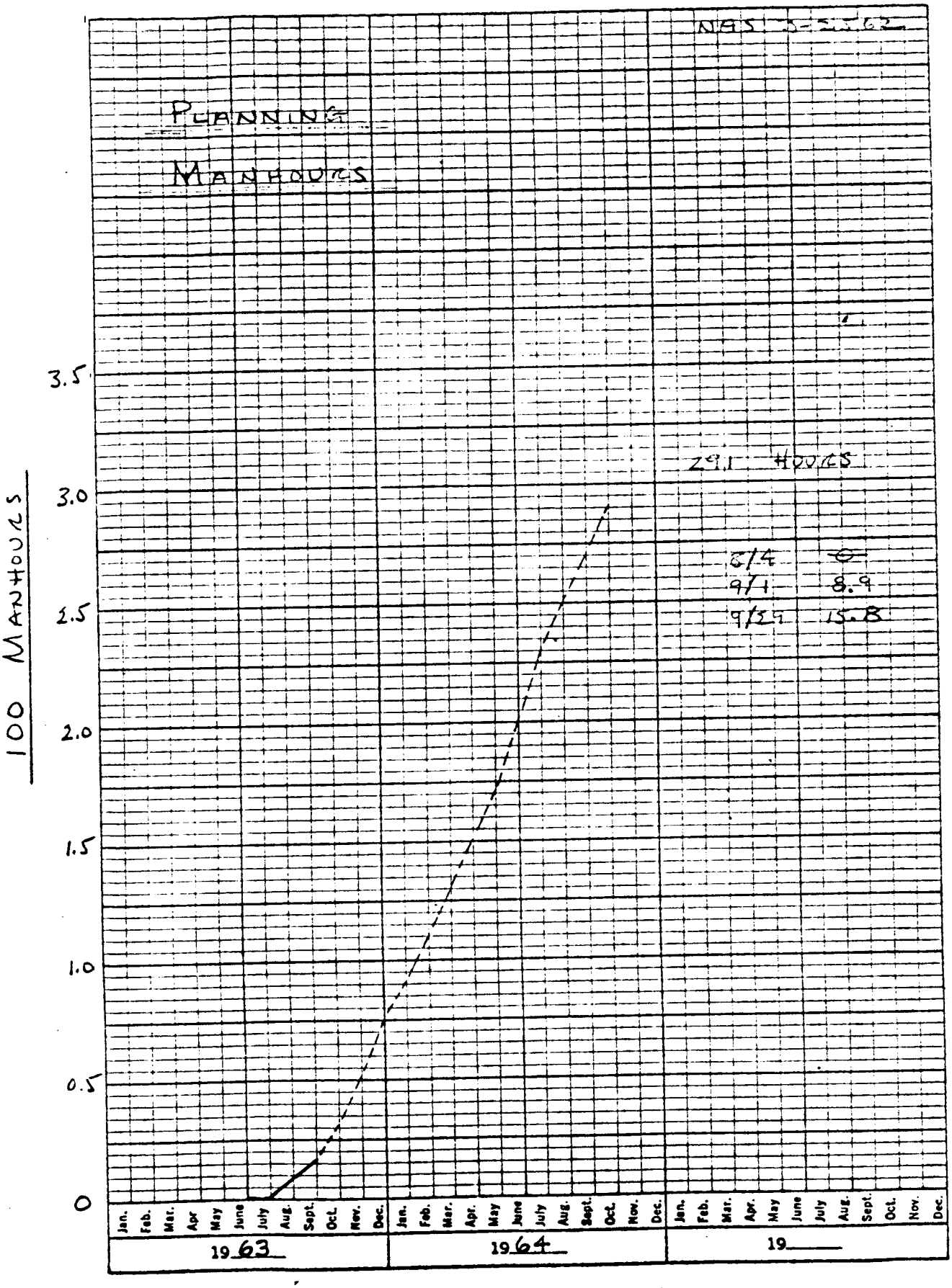

NAS 5-2162
PLANNING
MANHOURS
100 MANHOURS
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
291 HOURS
6/4 —
9/1 8.9
9/29 15.8
Jan. Feb. Mar. Apr May June July Aug. Sept. Oct. Nov. Dec.
1963
1964
19

FIGURE 11

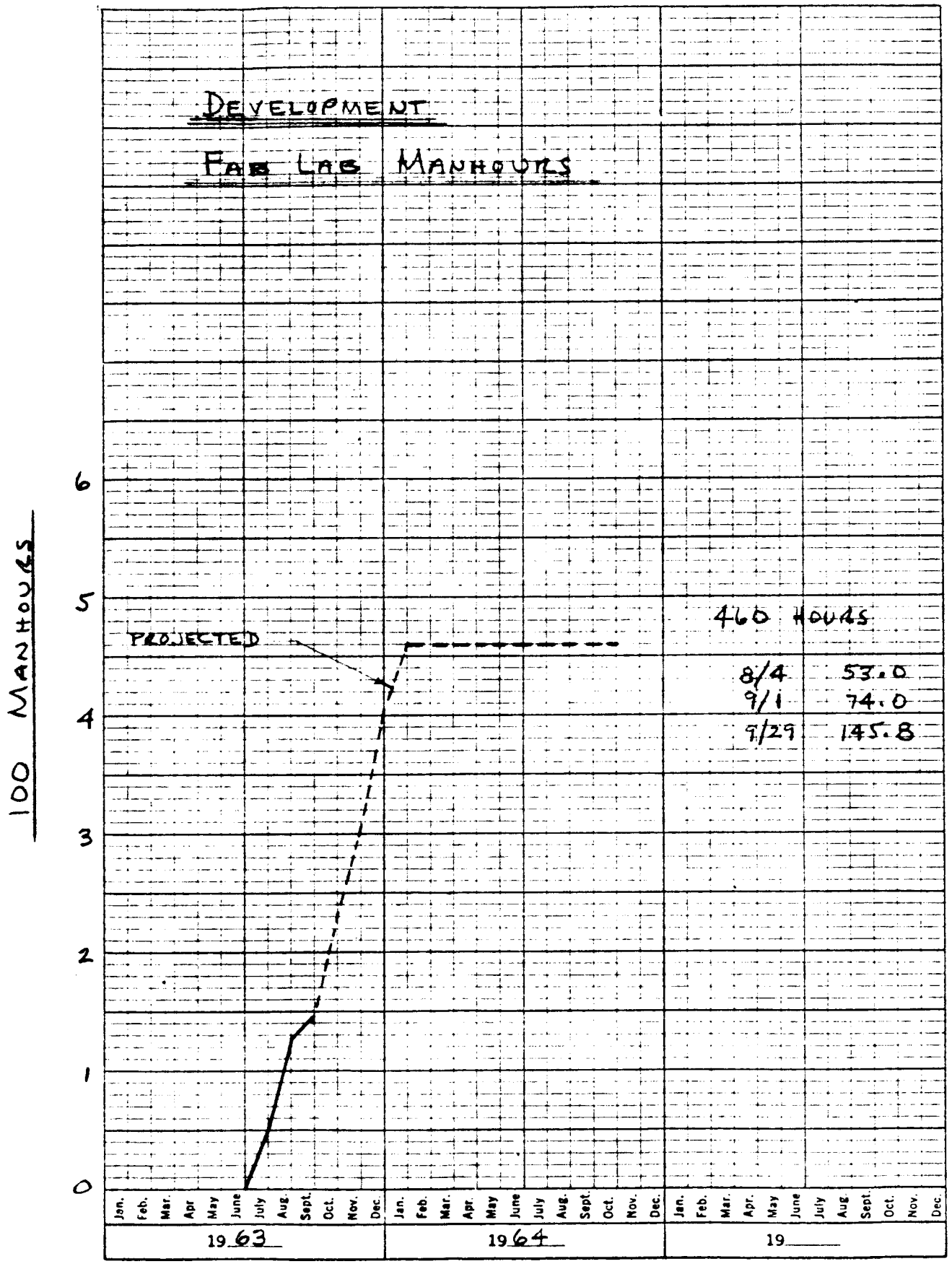

DEVELOPMENT
FAB LAB MANHOURS
100 MANHOURS
6
5
4
3
2
1
0
PROJECTED
460 HOURS
8/4 53.0
9/1 74.0
9/29 145.8
Jan. Feb. Mar. Apr May June July Aug. Sept. Oct. Nov. Dec.
1963
1964
19

FIGURE 12

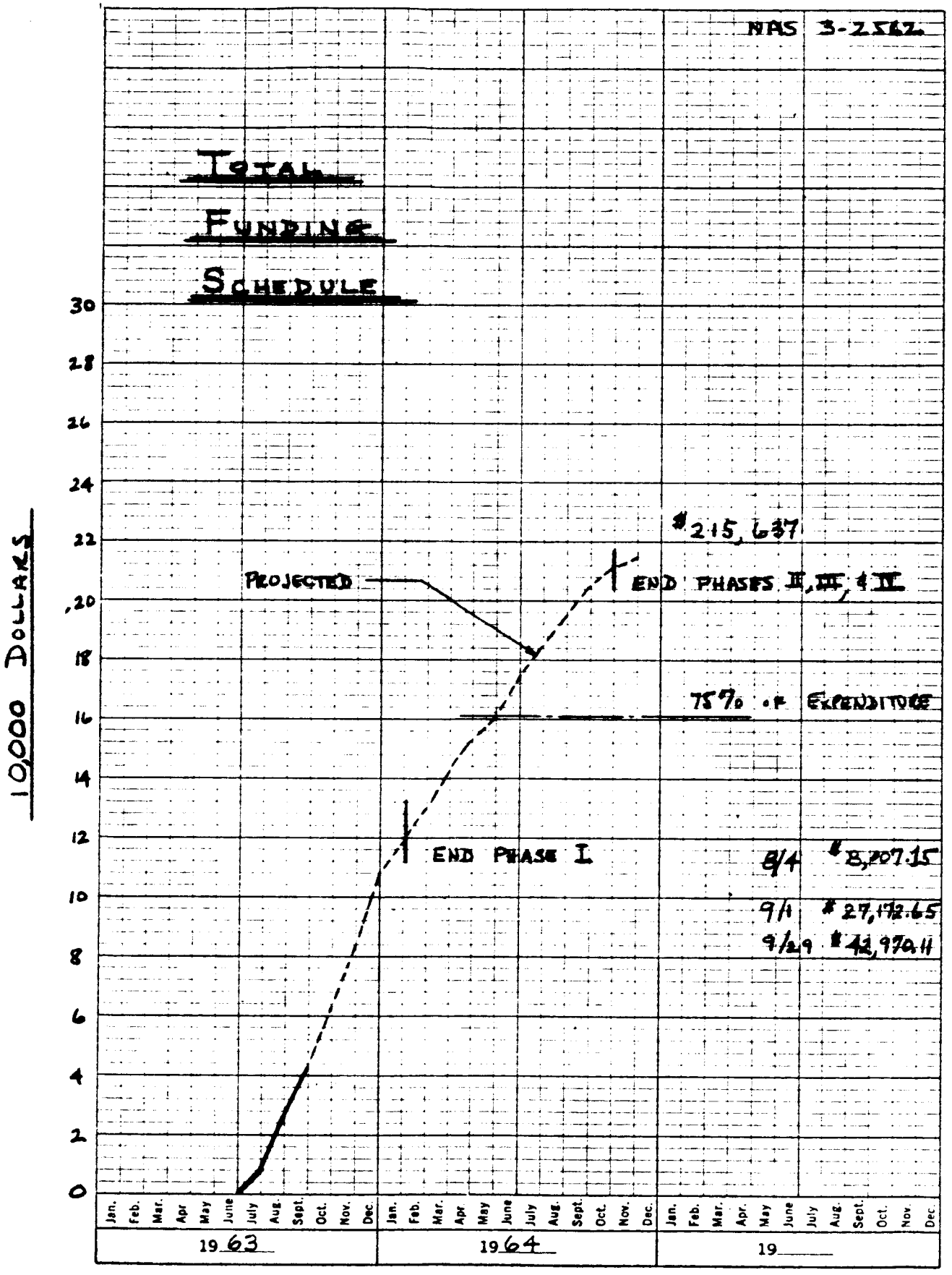

NAS 3-2562
TOTAL FUNDING SCHEDULE
10,000 DOLLARS
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
$215,637
PROJECTED
END PHASES II, III, & IV
75% OF EXPENDITURE
END PHASE I
8/4 $8,207.15
9/1 $27,172.65
9/29 $42,970.11
Jan. Feb. Mar. Apr May June July Aug. Sept. Oct. Nov. Dec.
1963
1964
19

FIGURE 13